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Fraunhofer-Institut fuer Chemische Technologie

Investigation of the Phase Stabilizing Effect of Potassium Fluoride on Ammonium Nitrate

R&D # 5955-CH-01-2
Purchase Order DAJA45-88-M-0051
Second Interim Report

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CONTENTS

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1. INTRODUCTION

In the first interim report the preparation of the samples, DSC-measurements and preliminary series of x-ray-diffraction measurements were reported.

This report will cover 5 series of diffraction measurements with dry samples of ammonium nitrate with a content of 2% KF.

series	temperature program
KF 230288	20/ 80/-70/ 80
KF 250288	20/ 80/-70/ 80
KF 230388	20/-70/ 80/-70
KF 170388	20/150/-70/ 20
KF 290388	20/-70/150/ 20

The documentation of the great amount of information provided by the x-ray diffraction measurements has been organized by means of an appendix. It contains for each series the original diffraction patterns, where each third patterns has been plotted, together with a difference curve, the explanation of which was included in the first interim report.

Besides, informations are given about the lattice distances d, the lattice parameters and the intensities of the different phases. These informations, however, are not yet available for all series, as the evaluation has not yet been fully completed.

Keywords: Graphs, West Germany, Phase Studies, Phase transformations, Transition temperature. (AW)

In the presentation and the discussion of the results it is referred to these informations in the appendix.

2. RESULTS OF THE MEASUREMENTS

2.1 SERIES KF 230288
20/ 80/-70/ 80

At room temperature the sample contains phase III with a small amount of Phase IV. The phase changes during the temperature program can be summarized as follows:

Heating 20 => 80:	III+IV --> III	end 40 °C
Cooling 80 =>-70:	III --> IV	begin 10 °C
	III --> V	begin 0 °C
	IV --> V	begin-32 °C
Heating-70 => 80:	V --> IV	begin -2 °C
	IV --> III	begin 35 °C

At the onset of the heating cycle the minor amount of phase IV disappears so that at 40 °C the whole sample consists of phase III. No further changes are observed on heating.

On cooling phase III changes into phase IV beginning at 10 °C. The transition III/IV not yet completed, surprisingly, the residue of III changes directly into phase V. Starting at -32 °C phase IV changes into phase V as expected. However, a small amount of the sample stays in phase IV even at the lowest temperatures indicated by a week $(111)_{IV}$-peak.

During the following heating cycle phase V changes completely into phase IV in the range of -2 to +10 °C, followed by a complete transition IV -> III between 35 and 45 °C.

The described transitions are visible in the difference curve in the appendix and can also be followed in the diffraction patterns by means of the peaks with the assigred indices.

2.2 SERIES KF 250288
20/ 80/-70/ 80

The sample was investigated with the same temperature program as in the series KF 230288. At the beginning the sample consists

again mainly of phase III with a small amount of phase IV. The phase changes can be summarized as follows:

Heating 20 => 80:	III+IV --> III	end	40 °C
Cooling 80 =>-70:	III --> V	begin	-10 °C
Heating-70 => 80:	V --> IV	begin	2 °C
	IV --> III	begin	37 °C

Immediately after beginning the heating cycle the minor amount of phase IV disappears as in the preceding series. On cooling, phase III changes completely into phase V starting at -10 °C. This direct and complete transition III/V had not been expected. In this detail the series distinguishes itself from the preceding one.

On heating, the complete transition V/III is followed by the complete transition IV/III.

The described transitions can again be followed in the diffraction patterns and with the difference curve. Additionally, the intensity curves of the peaks $(220)_{III}$, $(020)_{IV}$ and $(022)_{V}$ show nicely the appearing and disappearing phases. The intensity curves are rather rough. This is caused by the used monochromator, which provides high resolution on the expense of the intensities with the consequence of reduced measuring statistics.

The phase changes can also be followed by the lattice plane distances calculated from the appearing and disappearing peaks. The corresponding curves are included in the appendix. Intensive peaks appear earlier and disappear later during the observed phase transitions.

From the lattice plane distances of the different phases the lattice parameters of the elementary cell were calculated. A reasonable fit of the lattice parameters is considered a proof for the presence of the different phases. The plots are also included in the appendix. They also allow the determination of the temperature range, in which the phases are present. The parameter plots against temperature are performed with large increments of the y-axis so that the curves can be seen together

in one plot.

In three further separated plots the increments of the y-axis are smaller so that some more details are visible. The lattice parameters as well as the lattice plane distances do not show any irregular behaviour. If interest might arise to check the values in all details, still more sensitive curves and tables with the complete values can be provided.

2.3 SERIES KF 230388
20/-70/ 80/-70

In contrast to the preceding series the sample consists at room temperature mainly of phase IV with small amounts of phase III. The phase changes can be summarized as follows:

Cooling 20 => -70:	IV + III --> IV	end 5 - 0 °C
	IV --> V	begin -32 °C
Heating -70=> 80:	V --> IV	begin 0 °C
	IV --> III	begin 40 °C
Cooling 80 => -70:	III--> V	-15 °C

At the begin of the cooling cycle the small amount of phase III disappears followed by the transition IV/V. Again a small amount of phase IV is always present during the whole cooling cycle.

On heating the already known phase changes V/IV and IV/III are observed. Both transitions are completed in a relatively short temperature interval.

On cooling phase III changes directly into phase V in a short temperature interval, too.

There is no problem to follow the described changes in the diffraction patterns, whereas the difference curve doesn't show all changes clearly.

The appearing and disappearing phases again can be followed with the intensity curves, lattice distances and lattice parameter, as described earlier.

2.4 SERIES KF 170388

20/150/-70/ 20

At room temperature the sample consisted of mainly phase III with a minor amount of phase IV. The following phase changes were observed:

Heating 20 => 150:	III+IV --> III	end 50 °C
	III --> II	100 °C
	II --> I	130 °C
Cooling 150=> -70:	I --> II	130 °C
	II --> III	40 °C
	II --> IV	40 °C
	IV --> V	-25 °C
Heating -70=> 20:	III --> IV	-60 - 0 °C
	V --> IV	begin 0 °C

On heating the peaks of phase IV are detectable until 50 °C, before it changes completely into phase III. The transition III/II begins at 100 °C before the transition II/I at 130 °C.

On cooling, after the change I/II a simultaneous transition II/III and II/IV occurs, which was unexpected after the former measurements. At -25 °C phase IV changes into phase V, which is considered as a normal phase behaviour. Unexpectedly, phase III is present during the whole cooling cycle. It stays on, until it decreases during the heating cycle up to 0 °C, when the change V/IV begins.

The described phase changes can be followed in the diffraction patterns and in the difference curve. The intensity and lattice parameter curves were not yet available at the time of the report.

2.5 SERIES KF 290388

20/-70/150/ 20

At room temperature the sample consisted mainly of phase IV with a minor amount of phase III. The following phase changes were observed:

		begin of transition
Cooling 20 => -70:	III --> IV	-10 °C
	III --> V	-10 °C
	IV --> V	-35 °C
Heating-70 => 150:	V --> IV	5 °C
	IV --> III	35 °C
	III --> II	102 °C
	II --> I	132 °C
Cooling 150 => 20:	I --> II	130 °C
	II --> III	55 °C

On cooling the smaller amount of phase III changes simultaneously into the phases IV and V. This cannot be seen clearly in the diffraction patterns. The intensity curves, however, show a decreasing intensity of phase III and, simultaneously, an increase of phase IV and V, before the phase IV intensity decreases due to the change IV/V. Again, the (111) peak of phase IV is detectable in the diffraction patterns during the whole cooling cycle.

On heating the change V/IV begins at 5 °C followed by the change IV/III starting at 35 °C and the changes III/II and II/I at 102 and 132 °C, resp.

On cooling phase II reappears at 130 and phase III reappears at 55 °C. The diffraction patterns show strong texture effects after the change I/II influencing strongly the peak intensities. This effect is very often observed on cooling, when the transition temperature II/I was surpassed.

3. DISCUSSION

Samples at room temperature

The samples had been stored for more than one week at room temperature before the measurements. They consist of a mixture of the phases IV and III. In different samples both phases can prevail.

The existence of phase III is due to the incorporation of potassium ions. The potassium concentration is not yet high enough to make phase III the only stable phase at room temperature.

Phase changes on heating

To get a more comprehensive view of the results, the phase changes have been summarized in table 1. It can be seen that on cooling from -70 °C phase V changes in all cases at about 0 °C into phase IV, which changes into phase III between 35 and 40 °C. The latter transition occurs in the same temperature range, when the heating starts at room temperature.

Heating up to 80 °C no further transition is observed. With a temperature program up to 150 °C phase III changes into phase II at about 100 °C. The transition temperature is increased compared to pure humide ammonium nitrate due to the presence of the potassium ions.

Further heating changes the sample into phase I as expected. Eventually the transition temperature might be slightly higher than in pure ammonium nitrate, however, the resolution of our measurements with 2.5 °C measuring intervals is not high enough to answer this question.

Phase changes on cooling

Cooling down from 150 °C causes the samples to change at 130 °C into phase II as expected. Surprisingly no hysteresis of the transition temperature on heating and cooling is observed. In pure ammonium nitrate transition temperatures of about 120 °C are normally observed.

The following phase change II/III occurs at essentially lower temperatures compared to heating at 35 - 40 °C. The transition temperature is low enough to allow occasionally the direct phase change II/IV, which is observed in pure, absolutely dry ammonium

nitrate.

The further cooling again includes more than one transition path. The expected path consists of the phase change III/IV in an observed temperature interval from -10 to +10 °C. At these temperatures, however, the direct transition III/V at 0 to -15 °C is already possible.

In case of changing into phase IV the transition IV/V follows at temperatures from -25 to -40 °C, which is rather sluggish. This transition is not complete. Weak peaks of phase IV are detectable during the whole cooling cycle down to -70 °C. This phase change corresponds to the change in pure ammonium nitrate. Obviously, it was not influenced by the incorporated potassium ions.

Summary

A summary is given in table 2, which contains the different transitions paths, which were found, together with all observed transition temperatures. The table shows that the transitions II/III, III/IV and IV/V occur with strong hysteresis on heating and cooling.

The decreased transition temperatures on cooling obviously come so close to the stability ranges of the deep temperature phases that even direct phase changes II --> IV and III --> V are possible.

The measurements show, that the concentration of 2% of the incorporated potassium ions is not yet sufficient to exclude phase changes under storage conditions.

Table 1: Summarized Phase Changes

```
      20                    80                 -70                    80
          end 40             10      -32            -2       35
2302  |III+IV ->III  -|--> IV   --> V |  -->    IV -->    III  |
                          0   V

      20                    80                 -70                    80
          end 40             -10                     2       37
2502  |III+IV ->III  -|--> V        ----|--->   IV -->    III  |

      20                            150                          -70        20
          end 50  100     130        130     40                      0     0
1703  |III+IV ->III --> II --> I |  --> II  --> III   -->   |-->V  -->IV |
                                            40   IV   -25 V

      20                -70                               150               20
          -10   -35      5     35      102     132         130     55
2903  |IV+III ->IV --> V |-->IV -->III --> II  --> I |  --> II  -->III |
                V

      20                -70                   80      -70
          5-0   -32      0       40            -15
2303  |IV+III ->IV --> V |--> IV --> III |  -->  V |
```

Table 2: Observed transition paths and transition temperatures

```
                          heating

                    end 40, 40
    0, 5            end 40, 35          102          132
   -2, 2, 0         end 50, 35, 27      100          130

V                IV               III           II            I

   -25, -35           -10, 5             55          130
   -32, -32             0, 10            40          130

   -15,-10, 0
                           40

                          cooling
```

Appendix

A Peaklist

B Series KF230288

C Series Kf250288

D Series Kf170388

E Series Kf230388

F Series Kf290388

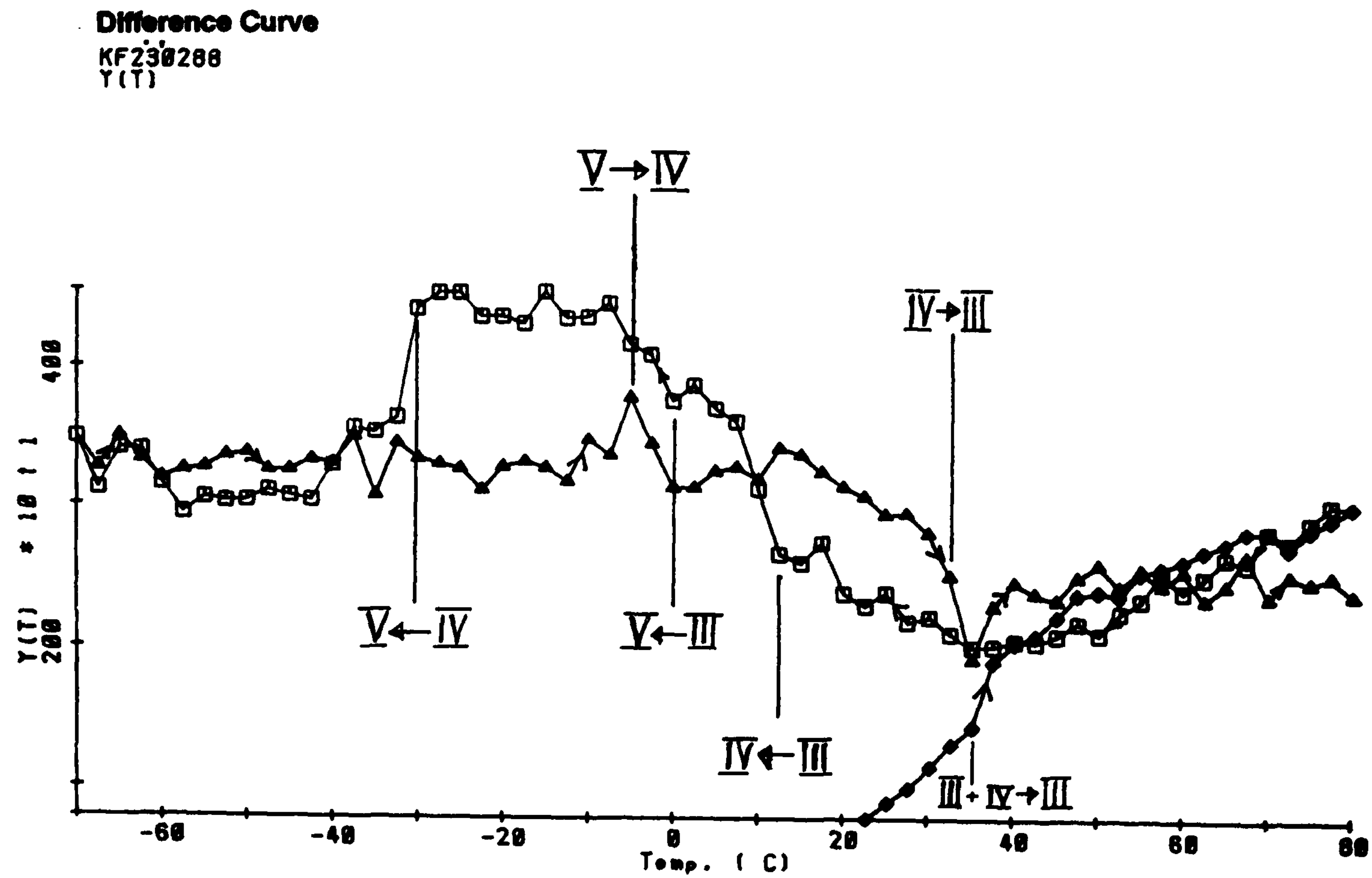
Peak List

Diffraction Angles of the Ammonium Nitrat Phases I,II,III,IV,V with Chromium Radiation.

Peak Nr.	2*Theta(ca.)	Phase	(hkl)
1	26.8	IV	$(001)_{IV}$
	27.05	V;II	$(001)_{II}$;$(002)_{V}$
2	29.2	III	$(101)_{III}$
3	30.5	I	$(100)_{I}$
4	33.0	II;V	$(020)_{V}$;$(110)_{II}$
5	33.67	V	$(200)_{V}$
6	33.8	IV	$(110)_{IV}$
7	34.24	III	$(111)_{III}$
8	39.53	III	$(120)_{III}$
9	41.43	III	$(210)_{III}$
10	41.78	III	$(021)_{III}$
11	43.5	II;V	$(111)_{II}$;$(022)_{V}$
12	43.8	I;IV	$(110)_{I}$;$(111)_{IV}$
13	45.7	III	$(121)_{III}$
14	46.9	IV	$(200)_{IV}$
15	46.9	V	$(212)_{V}$
16	47.72	II;V	$(200)_{II}$;$(220)_{V}$
17	47.72	III	$(211)_{III}$
18	49.8	V	$(221)_{V}$
19	50.-51.	IV	$(020)_{IV}$
20	51.85	III	$(220)_{III}$
21	53.2	II	$(210)_{II}$
22	53.8	V	$(130)_{V}$
23	55.0	IV	$(201)_{IV}$
24	55.7	V;II	$(002)_{II}$;$(004)_{V}$
25	56.54	III	$(130)_{III}$
26	57.4	IV	$(021)_{IV}$
27	58.5	V	$(014)_{V}$
28	59.02	III	$(022)_{III}$
29	60.41	III	$(310)_{III}$
30	60.9	III;II	$(211)_{II}$;$(202)_{III}$
31	60.9	IV	$(221)_{IV}$
32	61.2	V	$(132)_{V}$;$(312)_{V}$
33	61.8	IV	$(012)_{IV}$

Series KF 230288

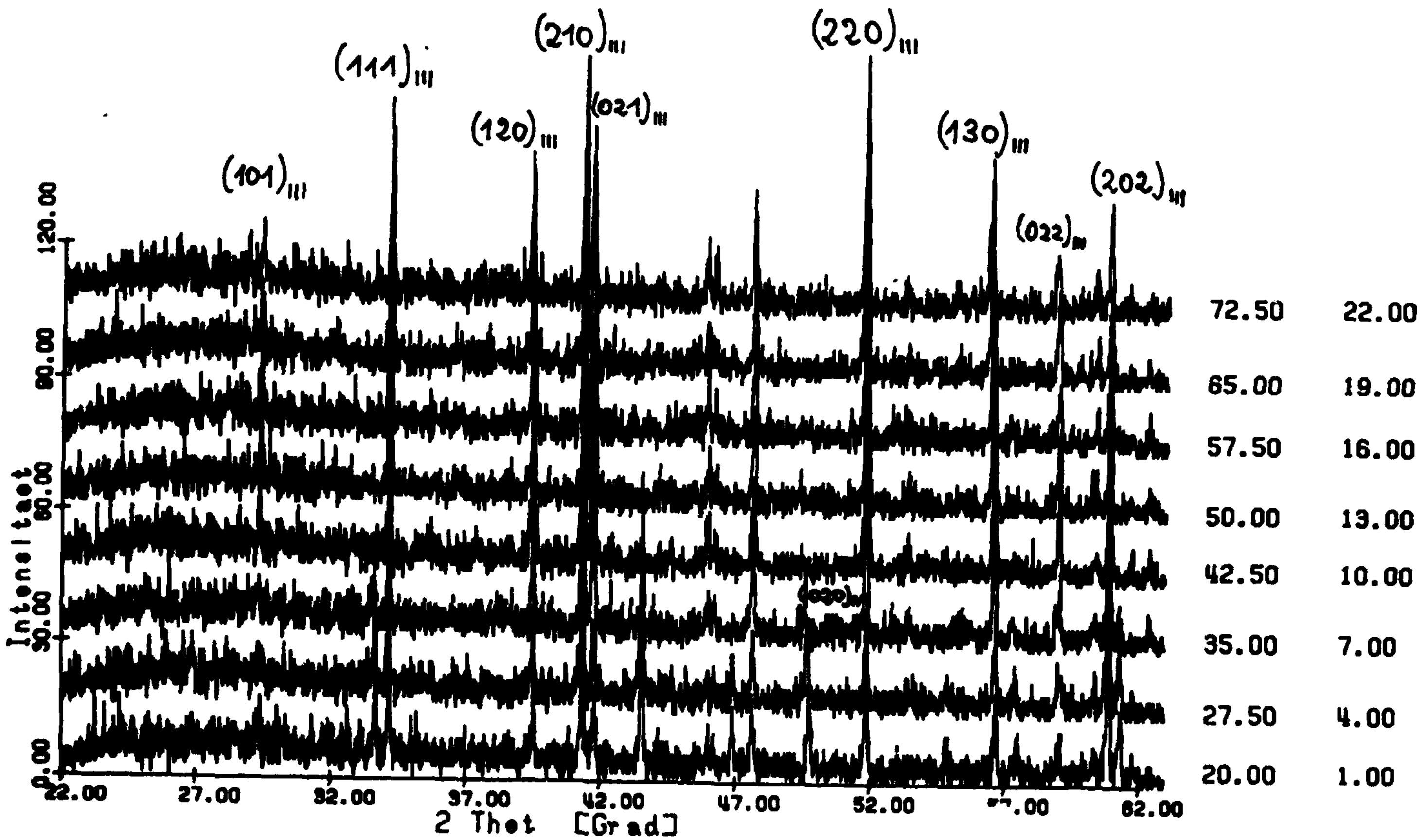
Temperature Program 20/80/—70/80


Difference Curve
KF230288
Y(T)
V→IV
IV→III
V←IV
V←III
IV←III
III+IV→III
400
200
Y(T) * 10 ! 1
-60
-40
-20
0
20
40
60
80
Temp. (C)

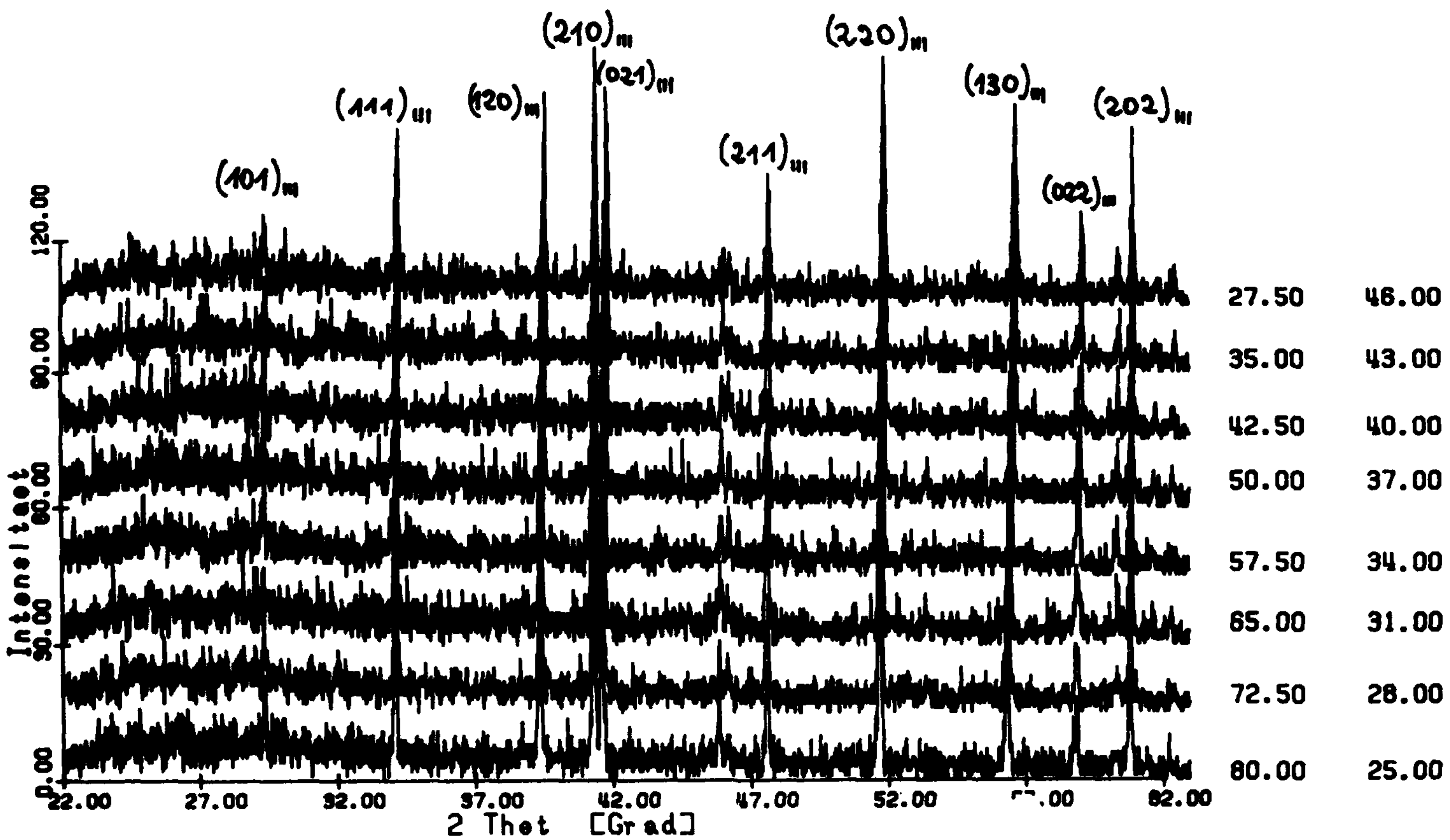
Diffraction Patterns

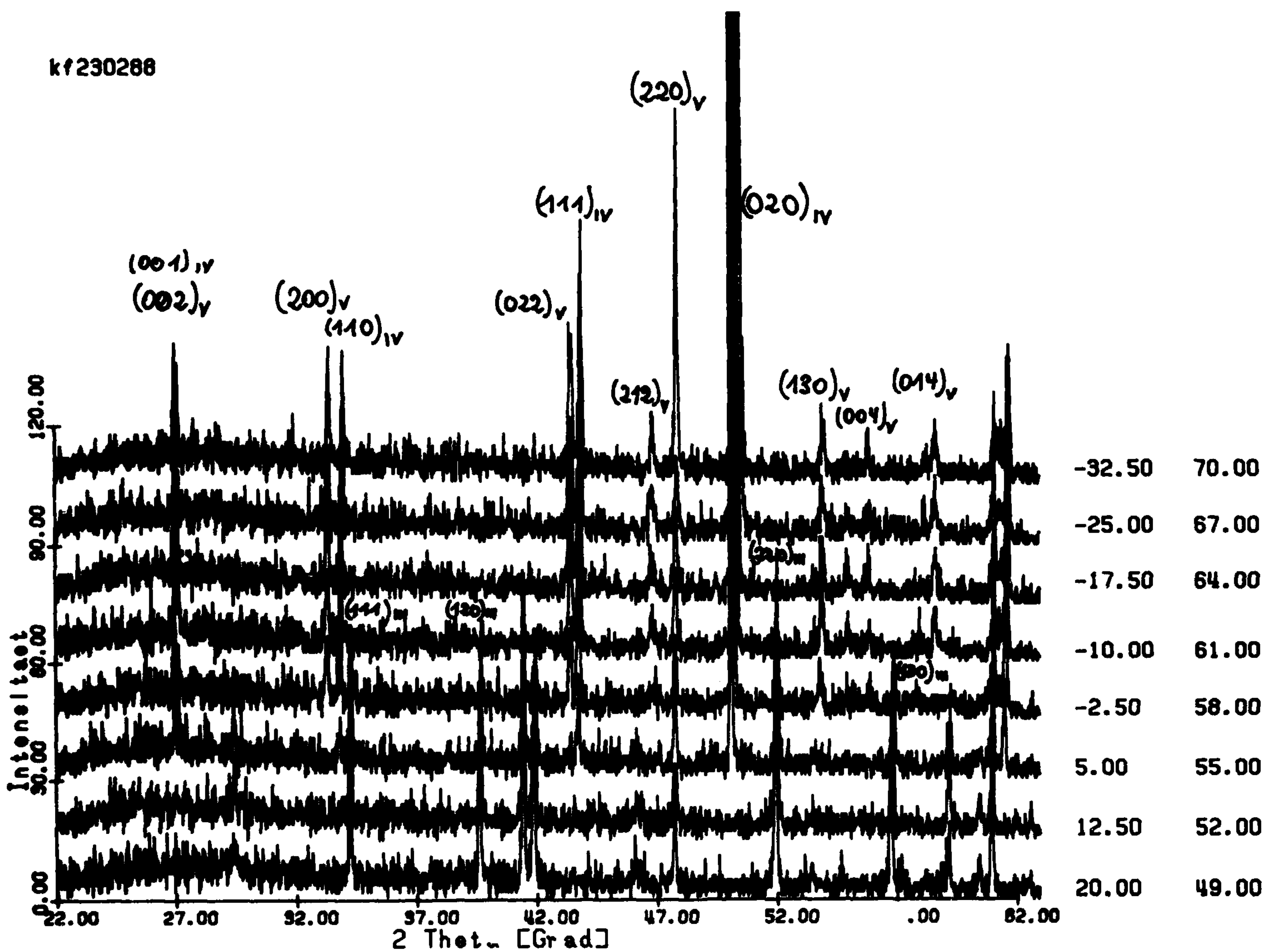
kf230288

20/80/-70/80

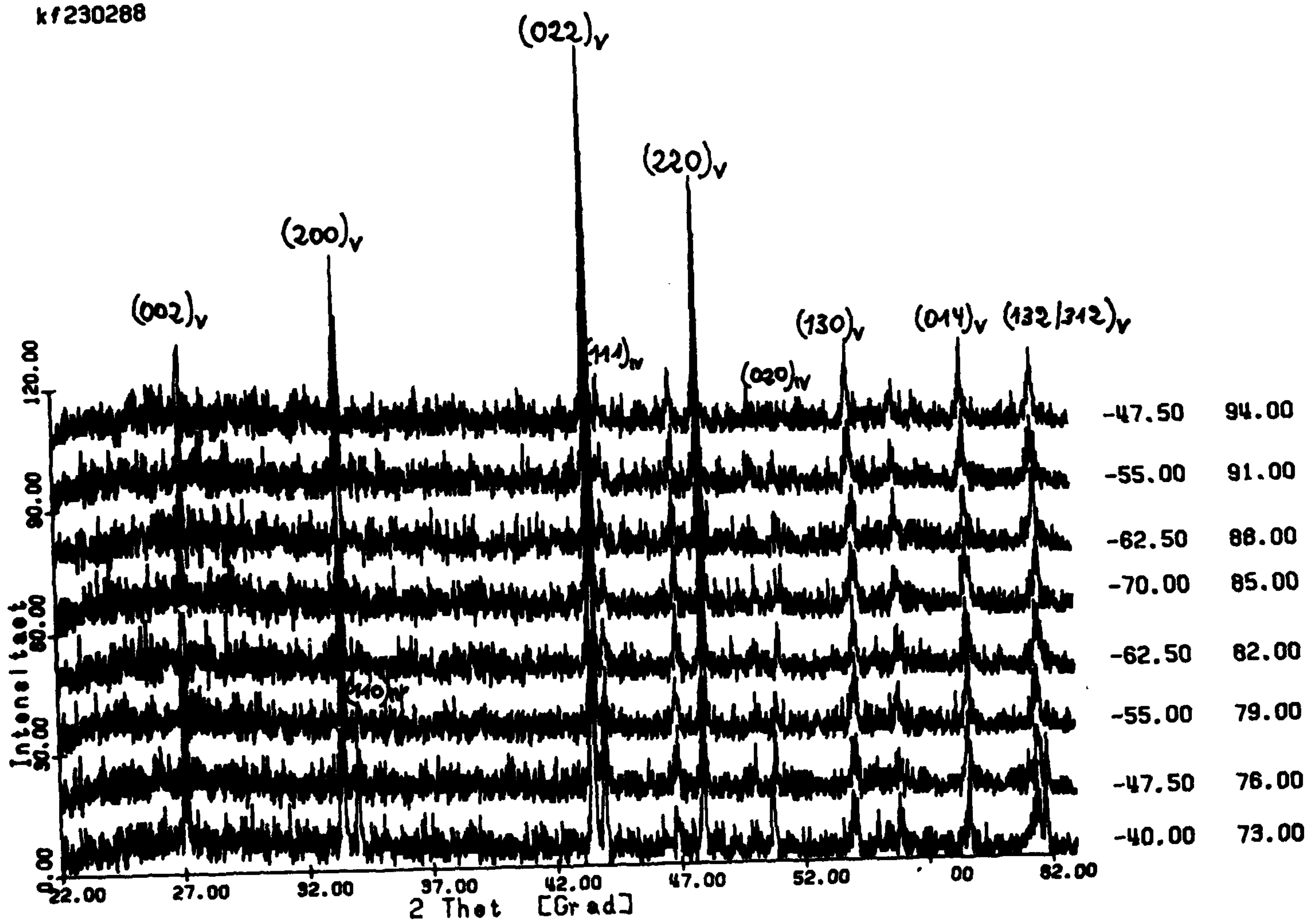


kf230288


(101)m
(111)m
(120)m
(210)m
(021)m
(211)m
(220)m
(130)m
(022)m
(202)m
Intensitaet
120.00
90.00
60.00
30.00
0.00
22.00
27.00
32.00
37.00
42.00
47.00
52.00
82.00
2 Thet [Grad]
27.50 46.00
35.00 43.00
42.50 40.00
50.00 37.00
57.50 34.00
65.00 31.00
72.50 28.00
80.00 25.00


kf230288
(001)IV
(002)V
(200)V
(110)IV
(022)V
(111)IV
(212)V
(220)V
(020)IV
(130)V
(004)V
(014)V
Intensitaet
120.00
90.00
60.00
30.00
0.00
22.00
27.00
32.00
37.00
42.00
47.00
52.00
.00
62.00
2 Theta [Grad]
-32.50 70.00
-25.00 67.00
-17.50 64.00
-10.00 61.00
-2.50 58.00
5.00 55.00
12.50 52.00
20.00 49.00

kf230288



(002)v
(200)v
(022)v
(111)v
(220)v
(020)v
(130)v
(014)v
(132/312)v
120.00
90.00
60.00
30.00
0.00
Intensitaet
22.00
27.00
32.00
37.00
42.00
47.00
52.00
00
62.00
2 Thet [Grad]
-47.50 94.00
-55.00 91.00
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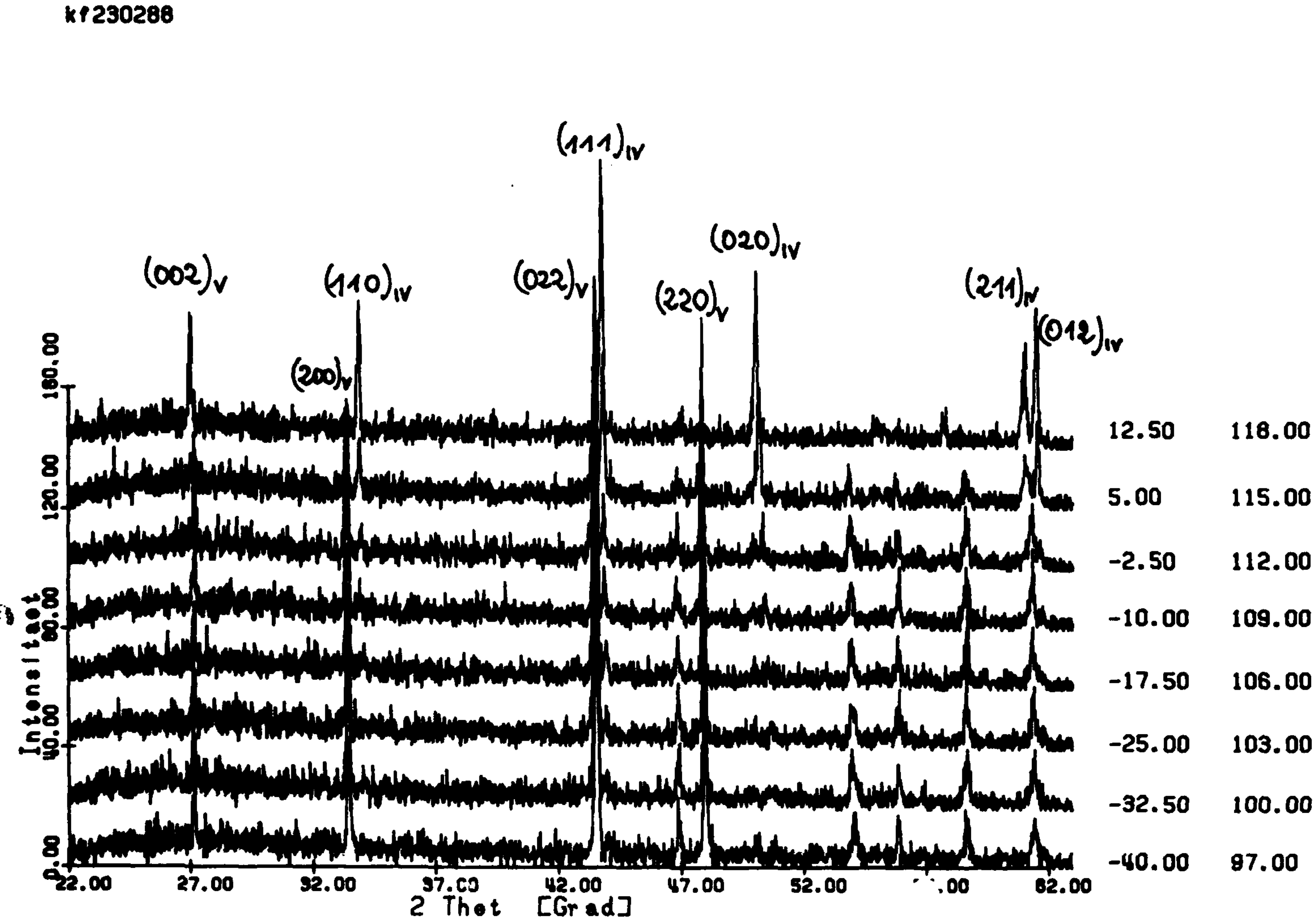
-47.50 76.00
-40.00 73.00


kf230288
(111)IV
(020)IV
(002)V
(110)IV
(022)V
(220)V
(211)IV
(012)IV
(200)V
180.00
120.00
80.00
40.00
0.00
Intensitaet
22.00
27.00
32.00
37.00
42.00
47.00
52.00
62.00
2 Thet [Grad]
12.50 118.00
5.00 115.00
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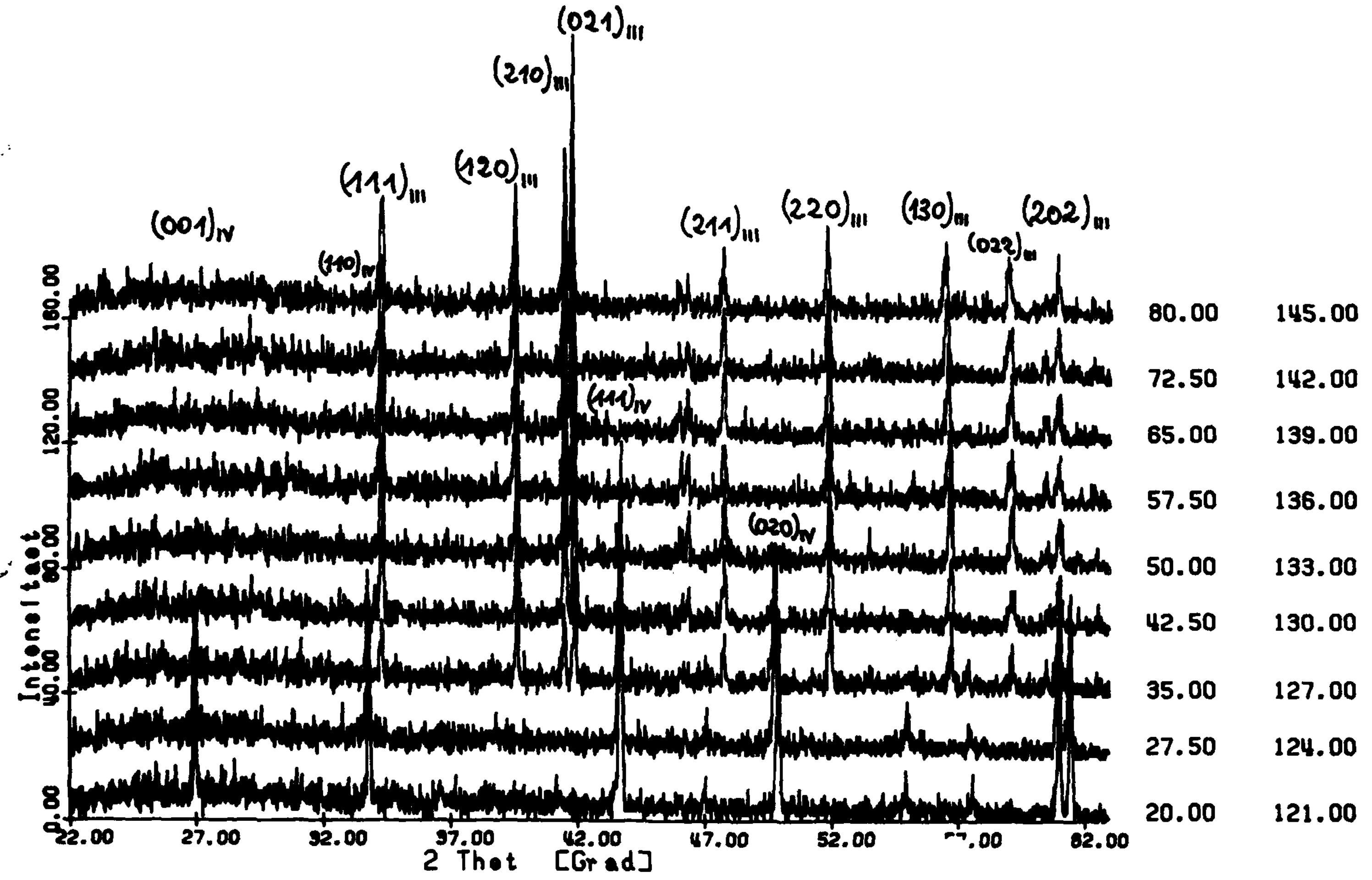
-40.00 97.00

kf230288



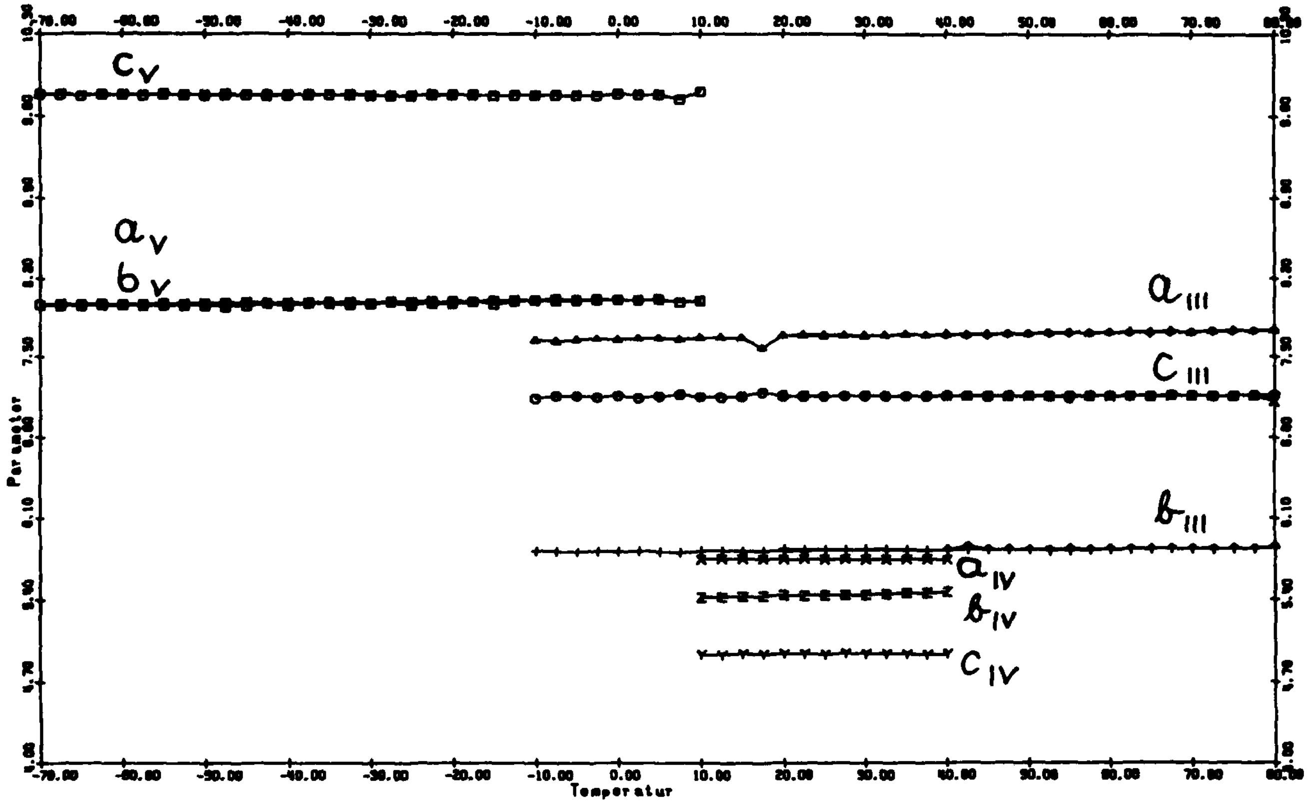
(001)IV
(110)IV
(111)III
(120)III
(210)III
(021)III
(111)IV
(211)III
(020)IV
(220)III
(130)III
(022)III
(202)III
Intensitaet
0.00
40.00
80.00
120.00
160.00
22.00
27.00
32.00
37.00
42.00
47.00
52.00
82.00
2 Thet [Grad]
80.00 145.00
72.50 142.00
65.00 139.00
57.50 136.00
50.00 133.00
42.50 130.00
35.00 127.00
27.50 124.00
20.00 121.00

Series
KF 250288

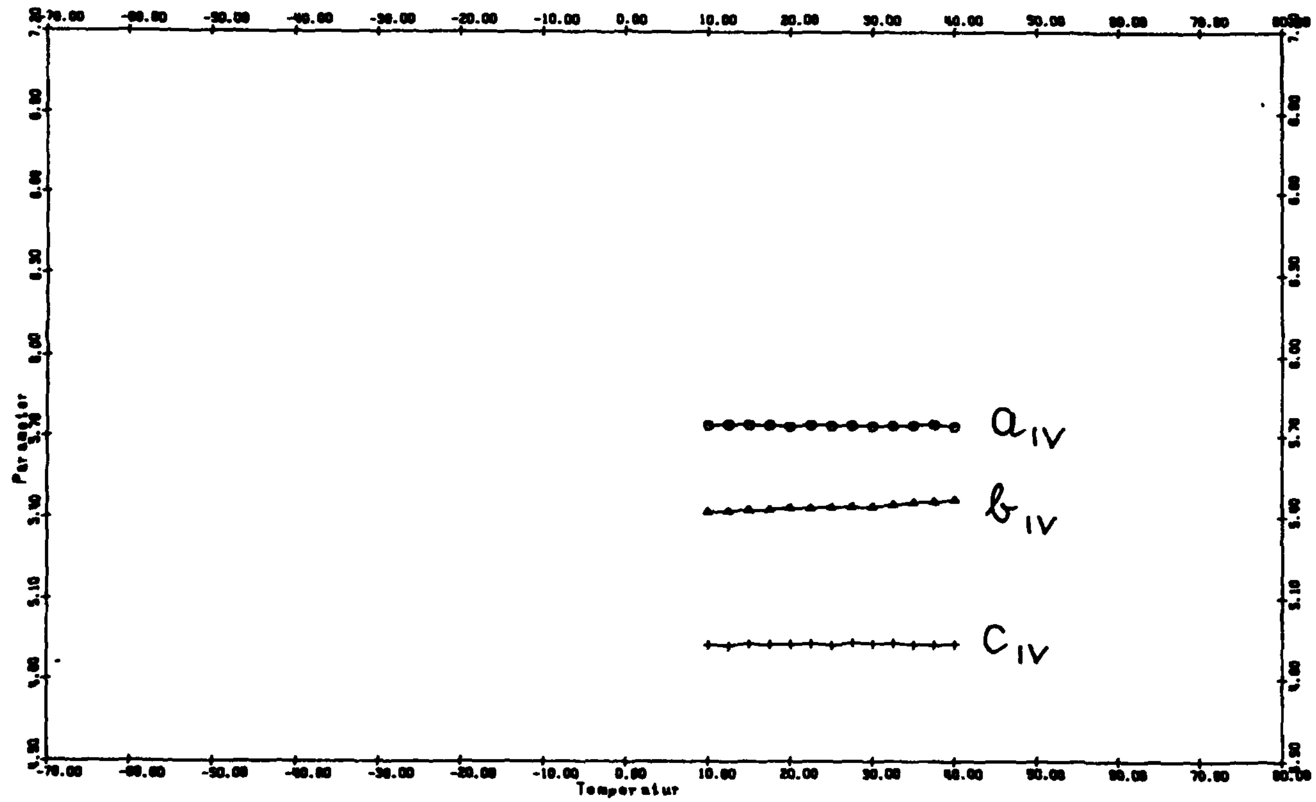
Temperature
Program
20/80/−70/80

KF 250288
20/80/-70/80

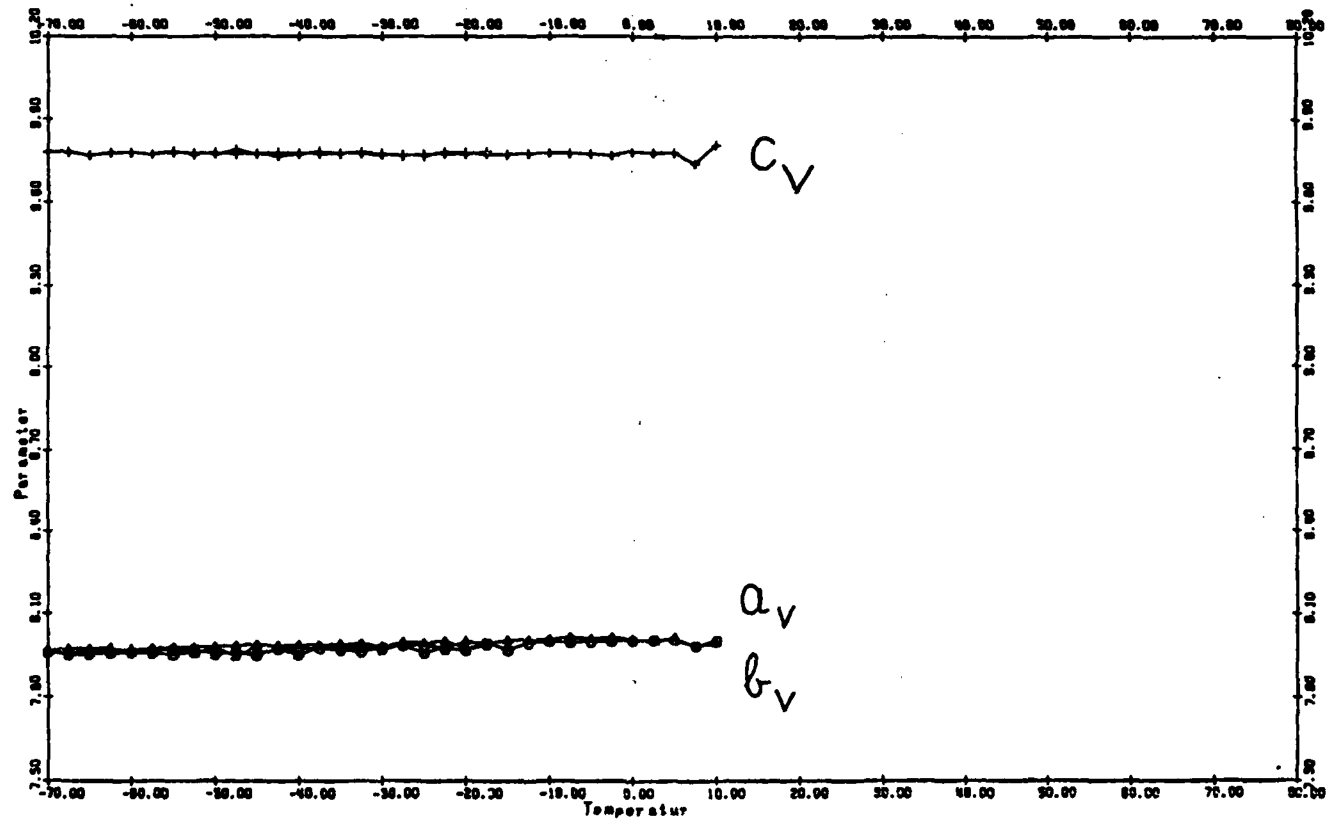
Lattice Parameters



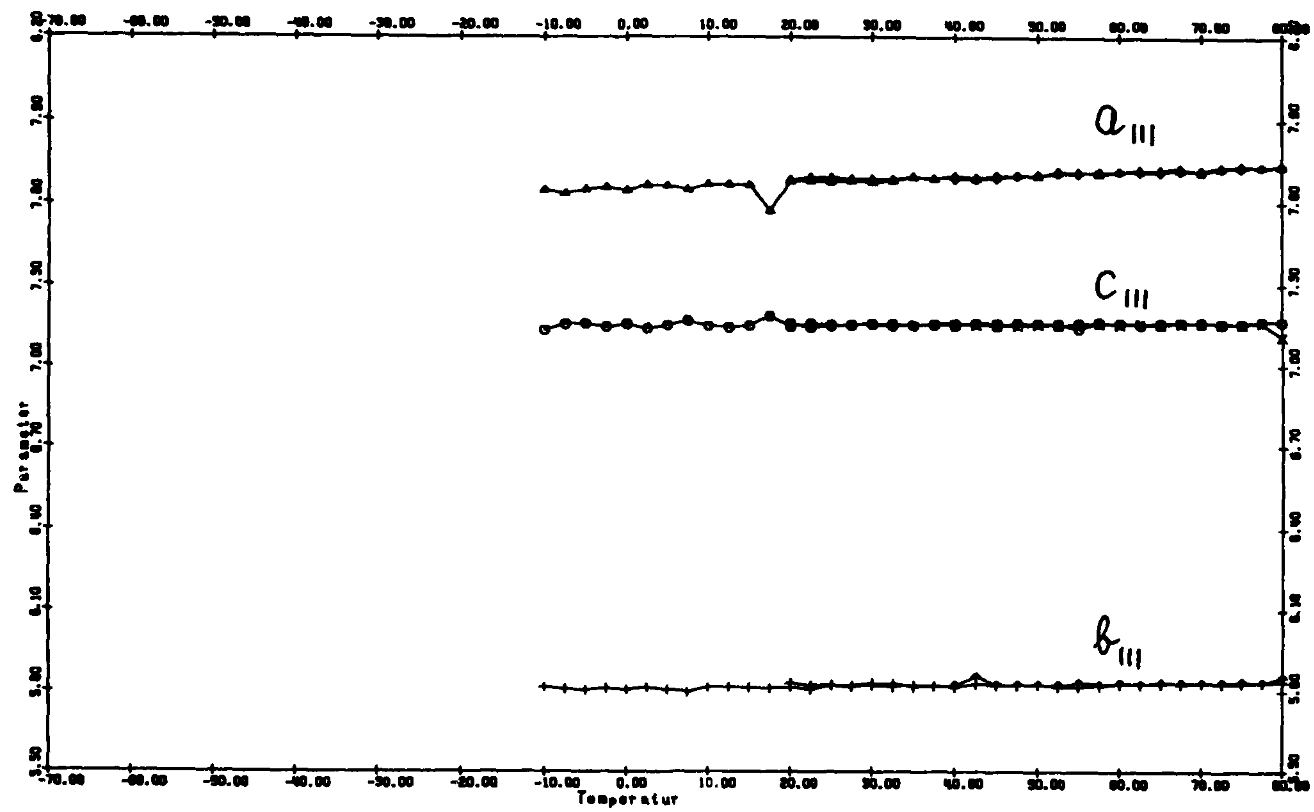
KF 250288
20/80/-70/80



KF 250238
20|80|-70|80

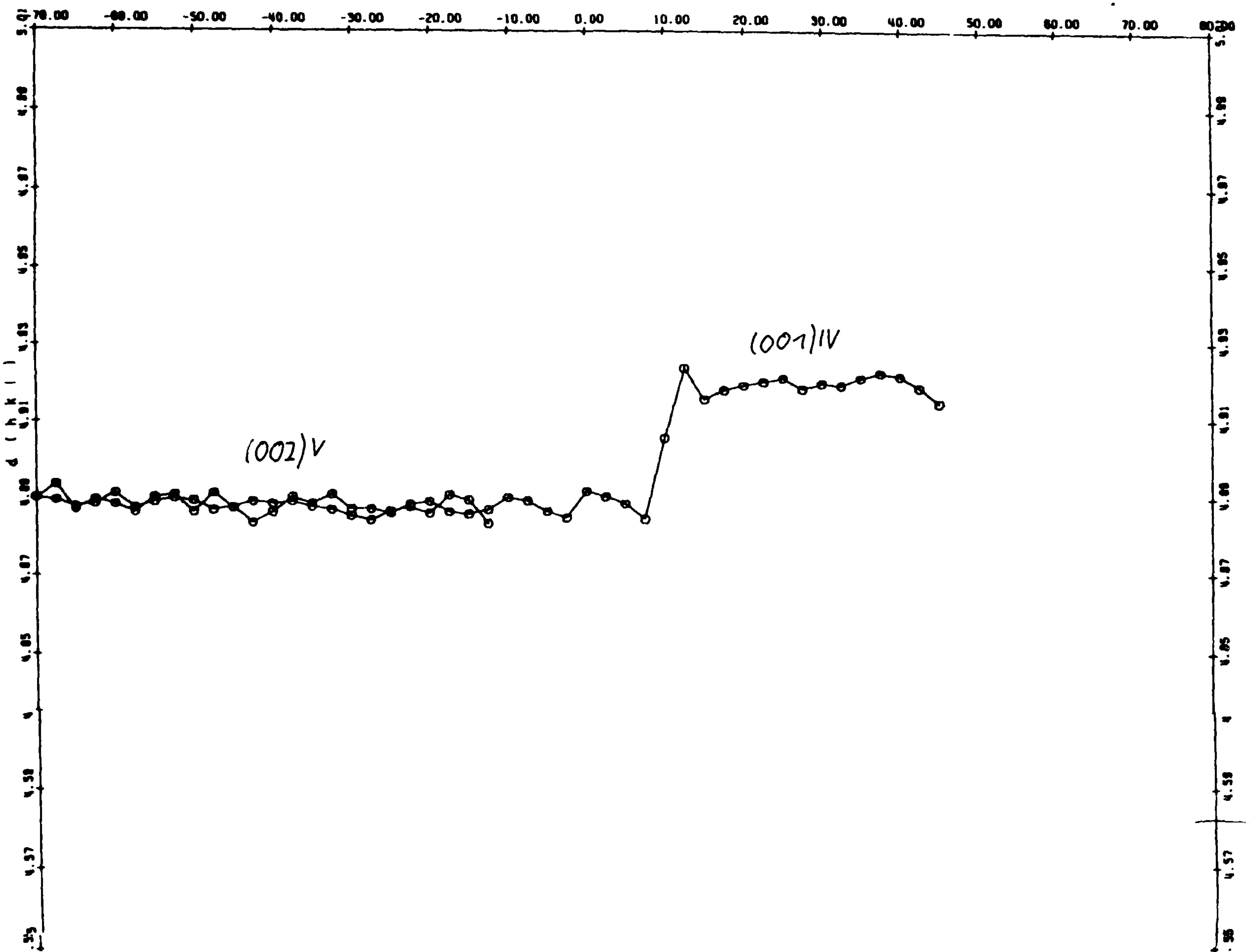


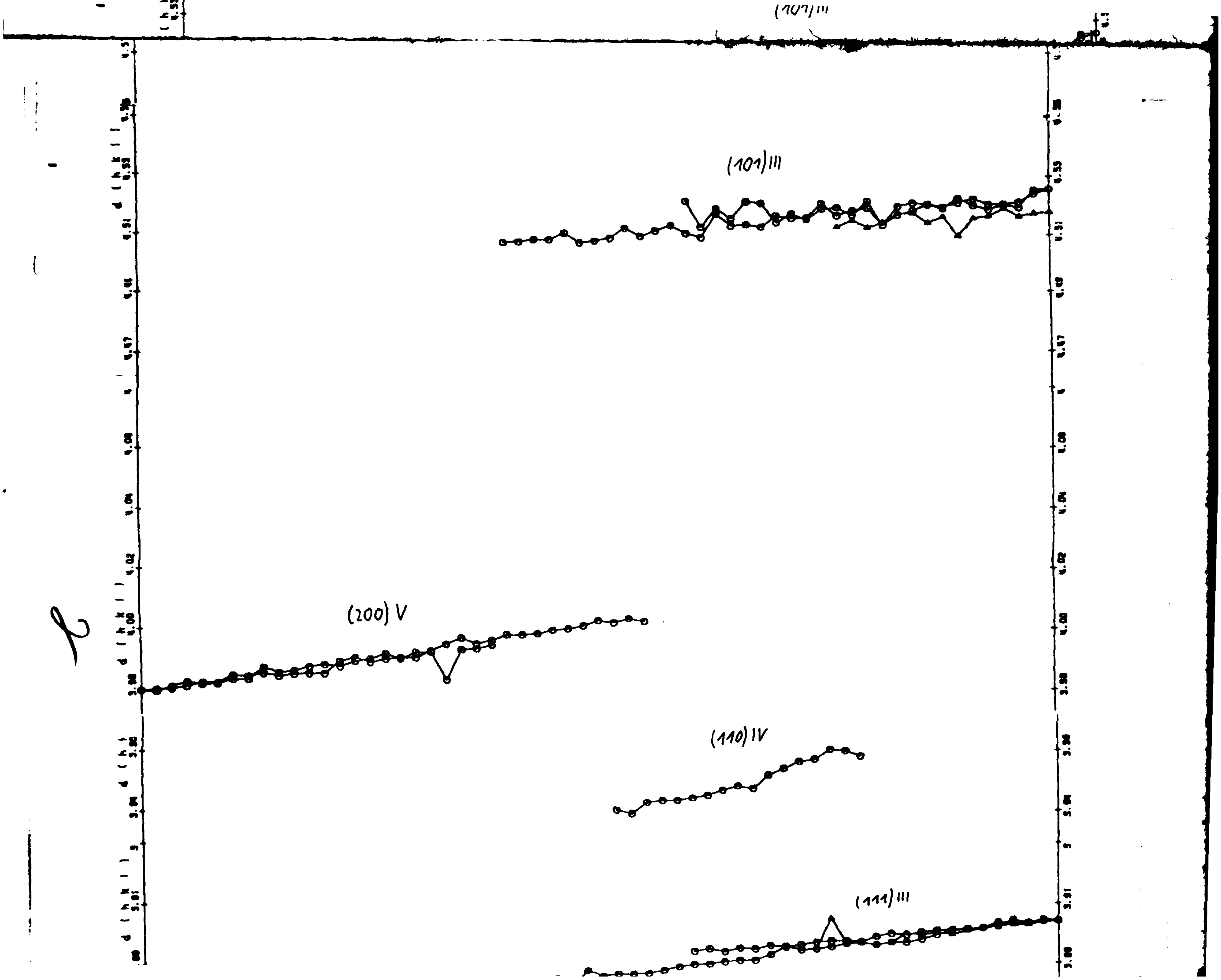
KF 250288
20/80/-70/80



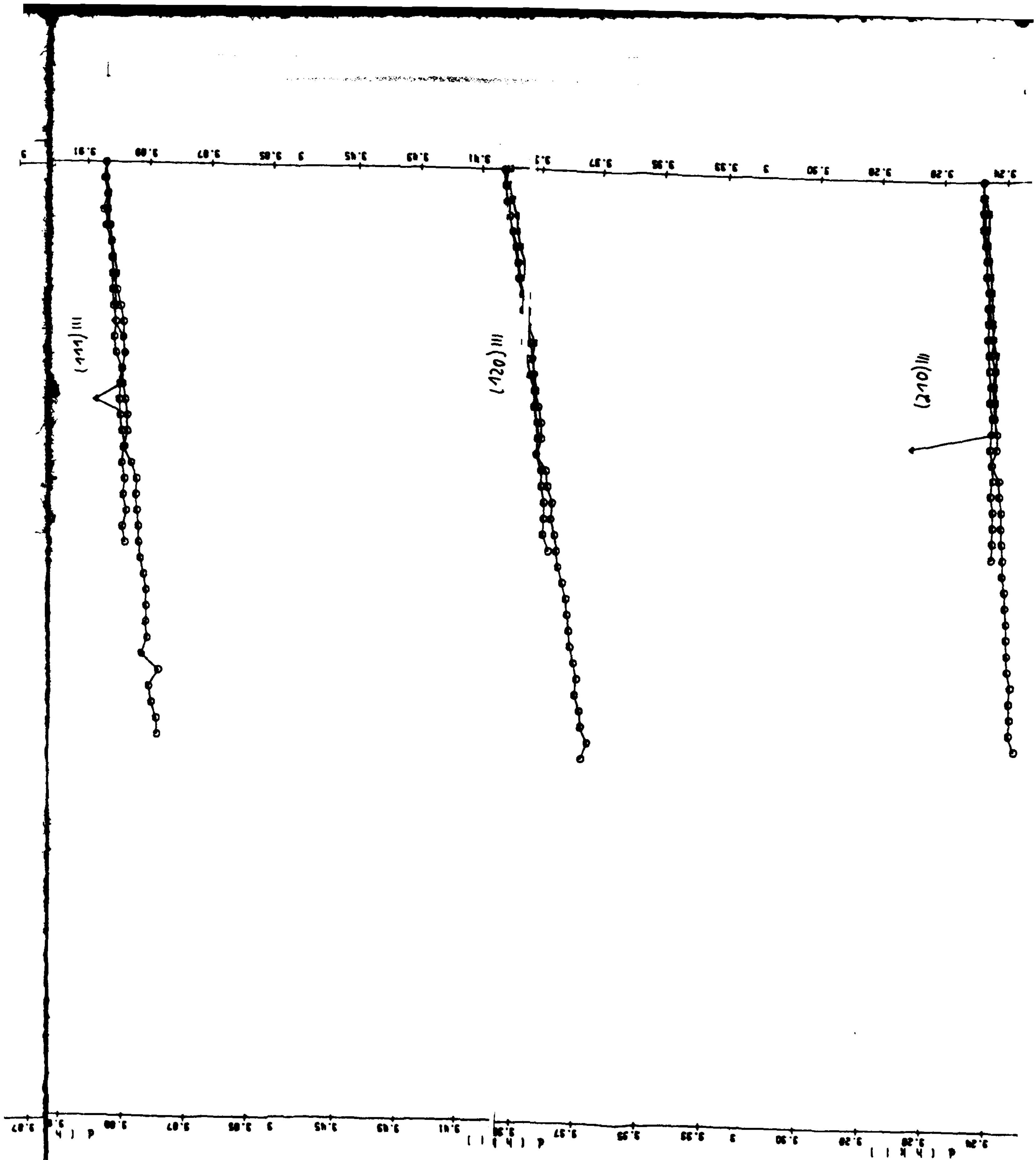
KF 250288

Lattice Distances (d)





(101) III
(200) V
(110) IV
(111) III
d (h k l)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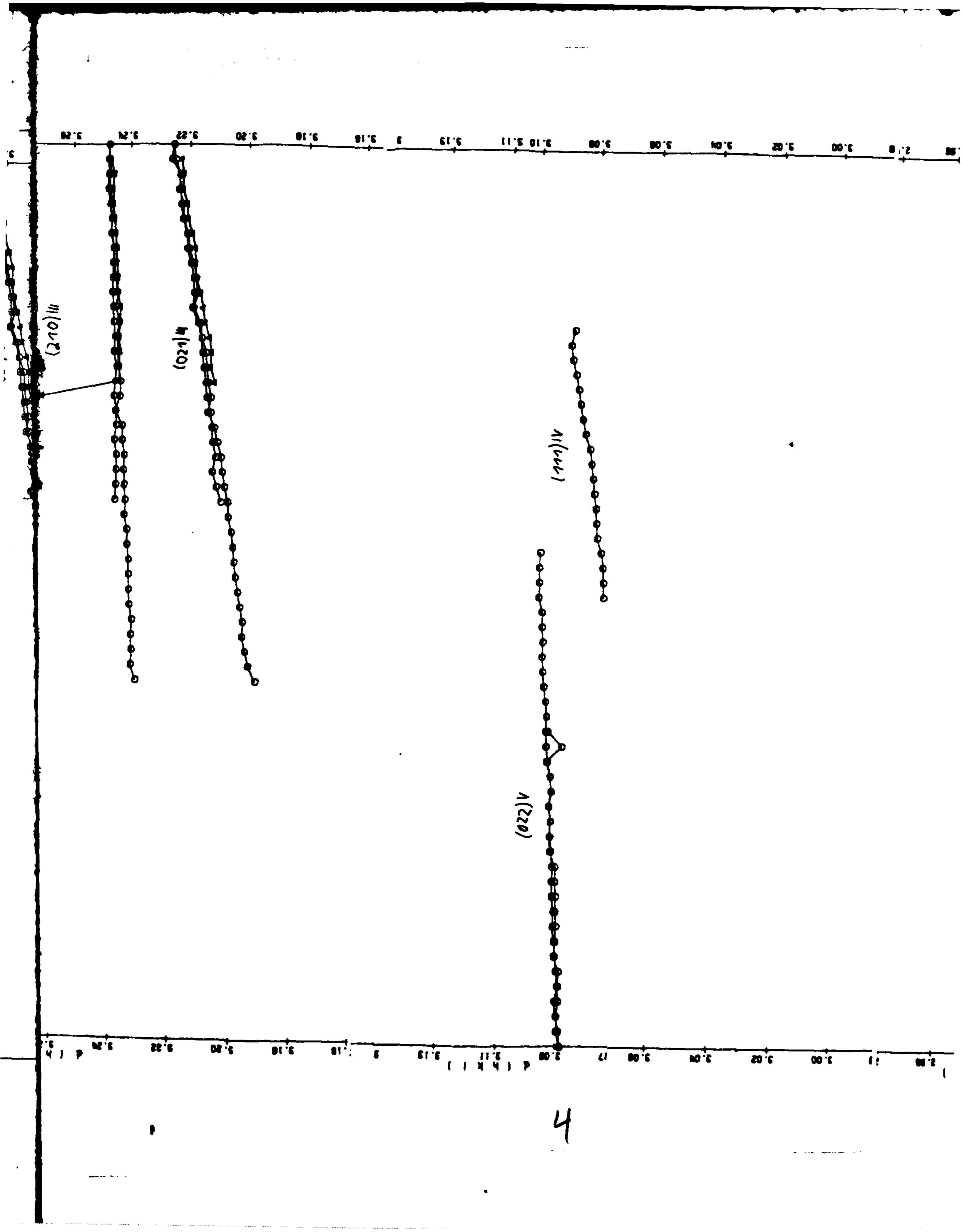
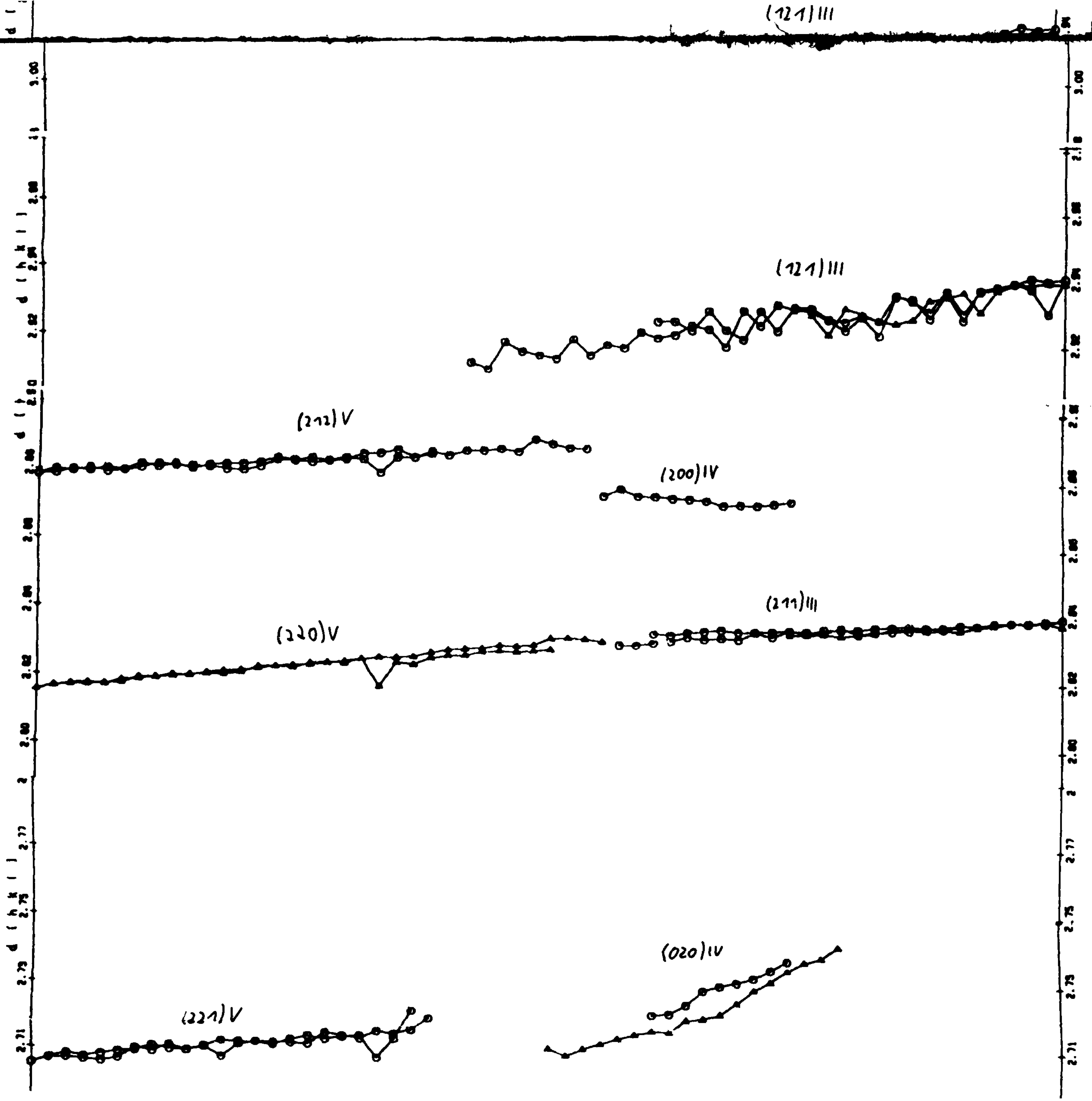
(120) III
(210) III

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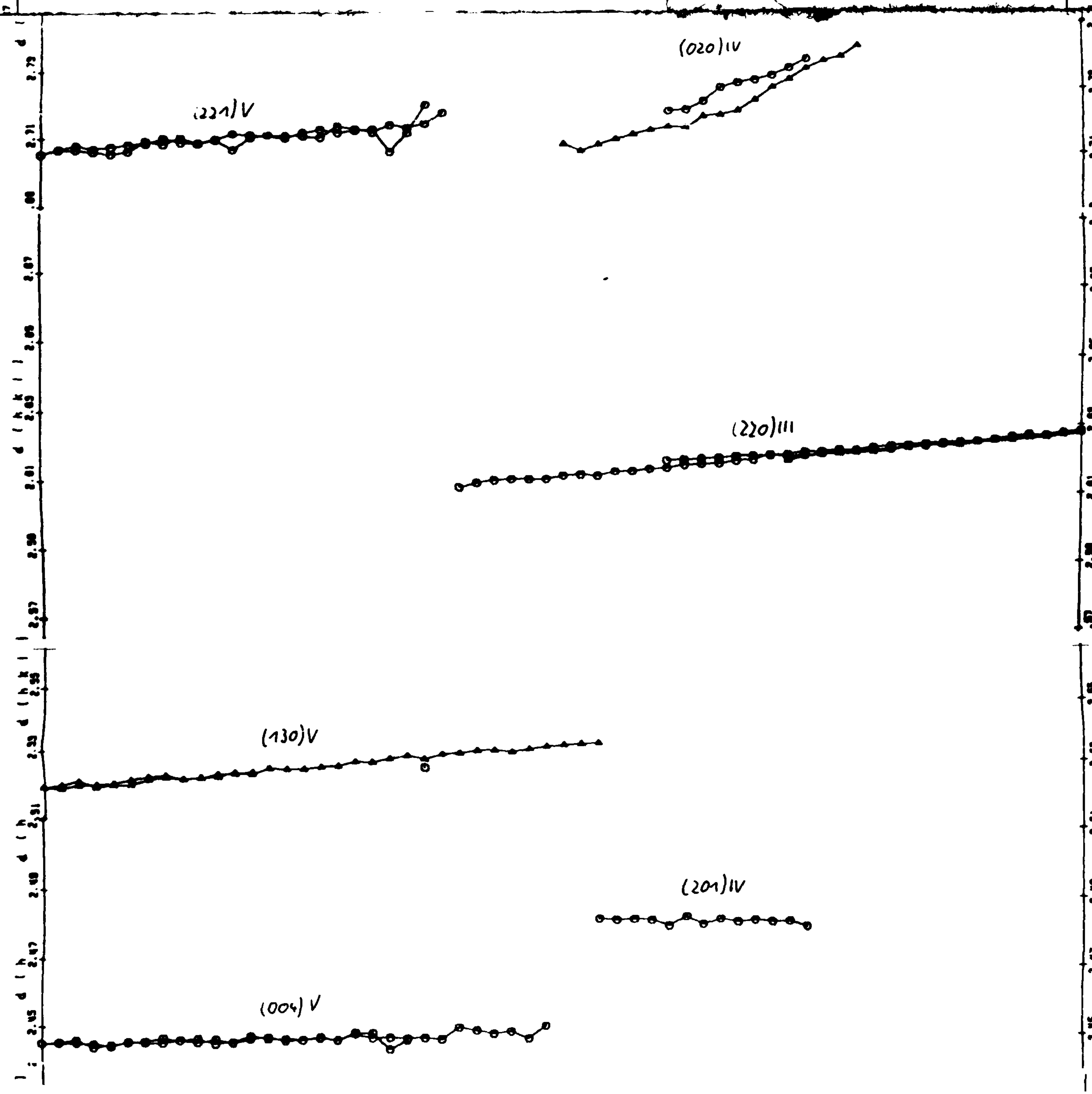
(200)IV
(211)III
(220)V
(020)IV
(221)V
d (h k l)



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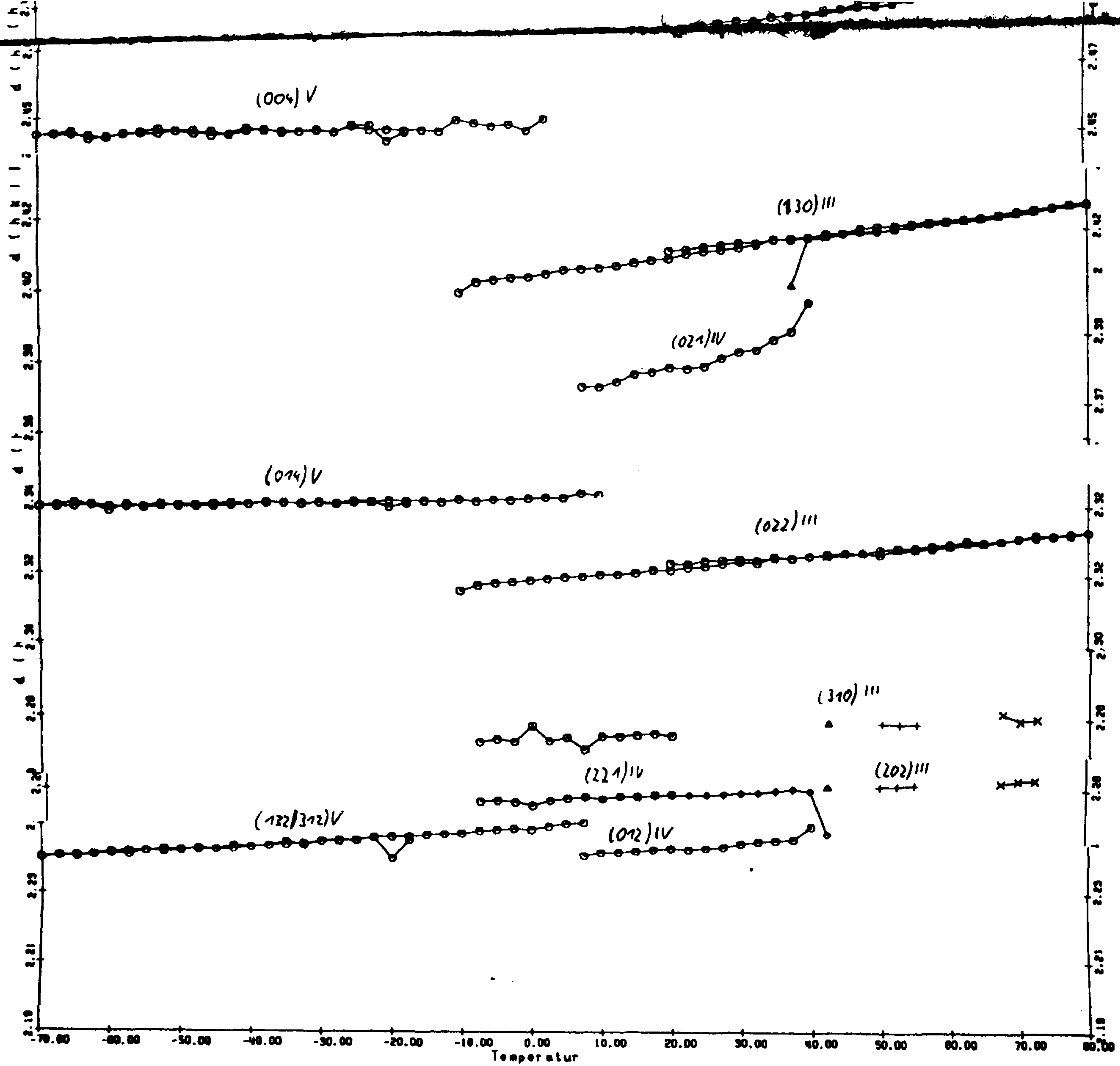
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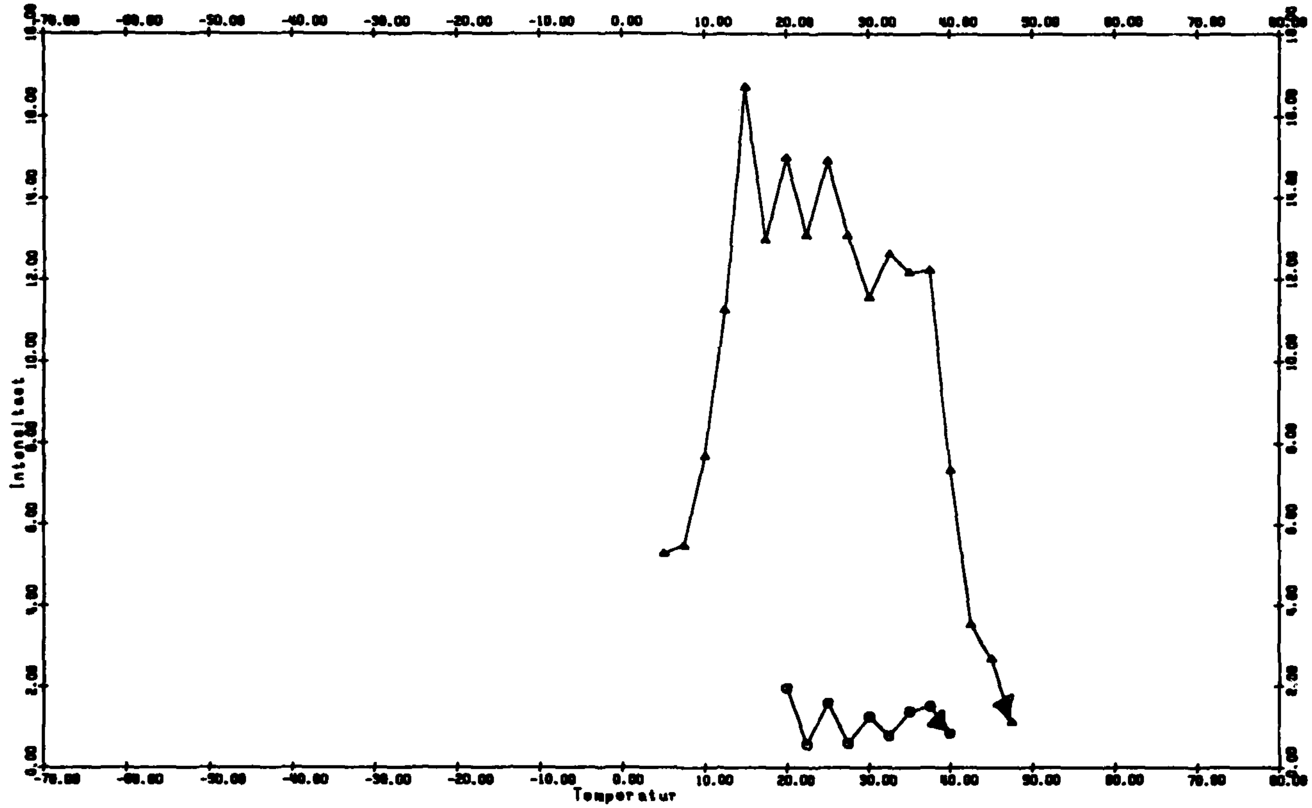
(130) III
(021) IV
(014) V
(022) III
(310) III
(221) IV
(202) III
(132/312) V
(012) IV
Temperatur
-70.00
-60.00
-50.00
-40.00
-30.00
-20.00
-10.00
0.00
10.00
20.00
30.00
40.00
50.00
60.00
70.00
80.00

7

kF 250288
$(020)_{IV}$

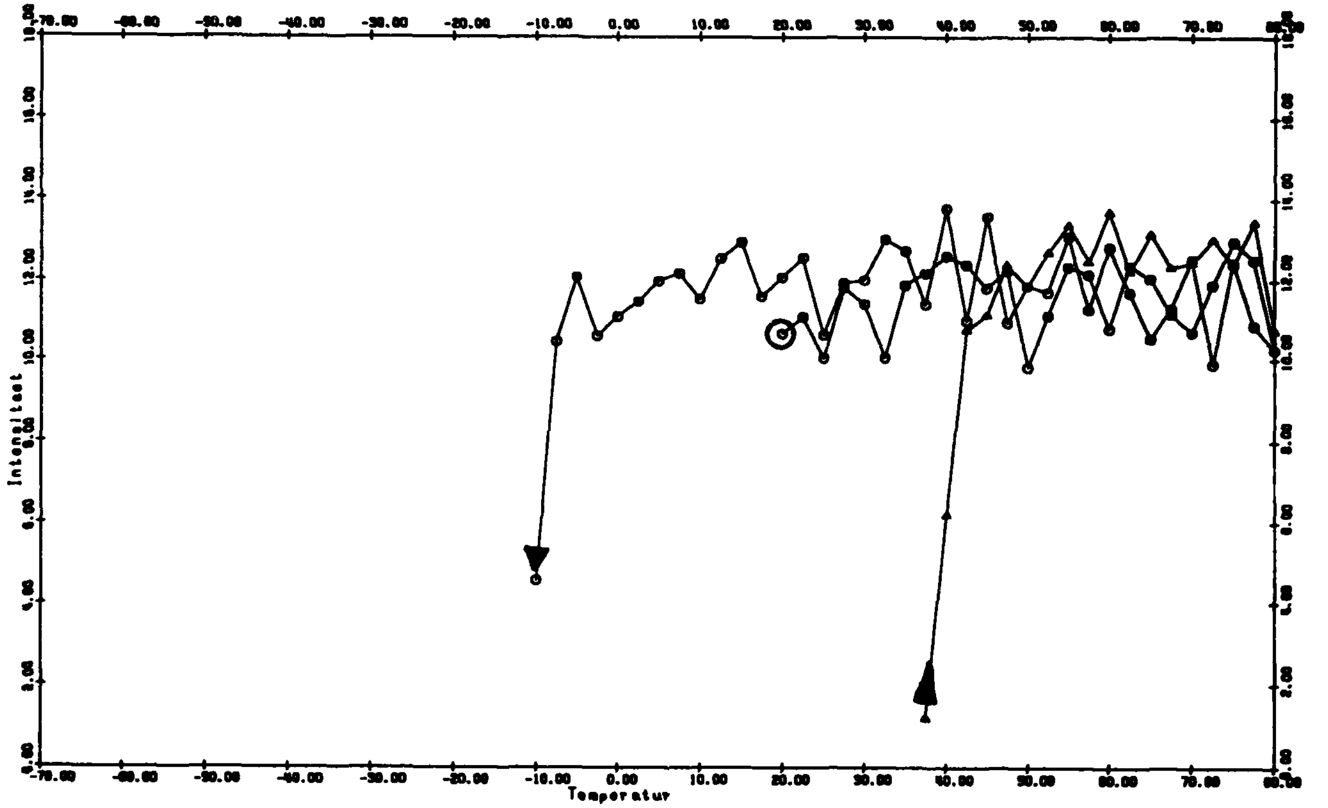
Intensities



KF 250288

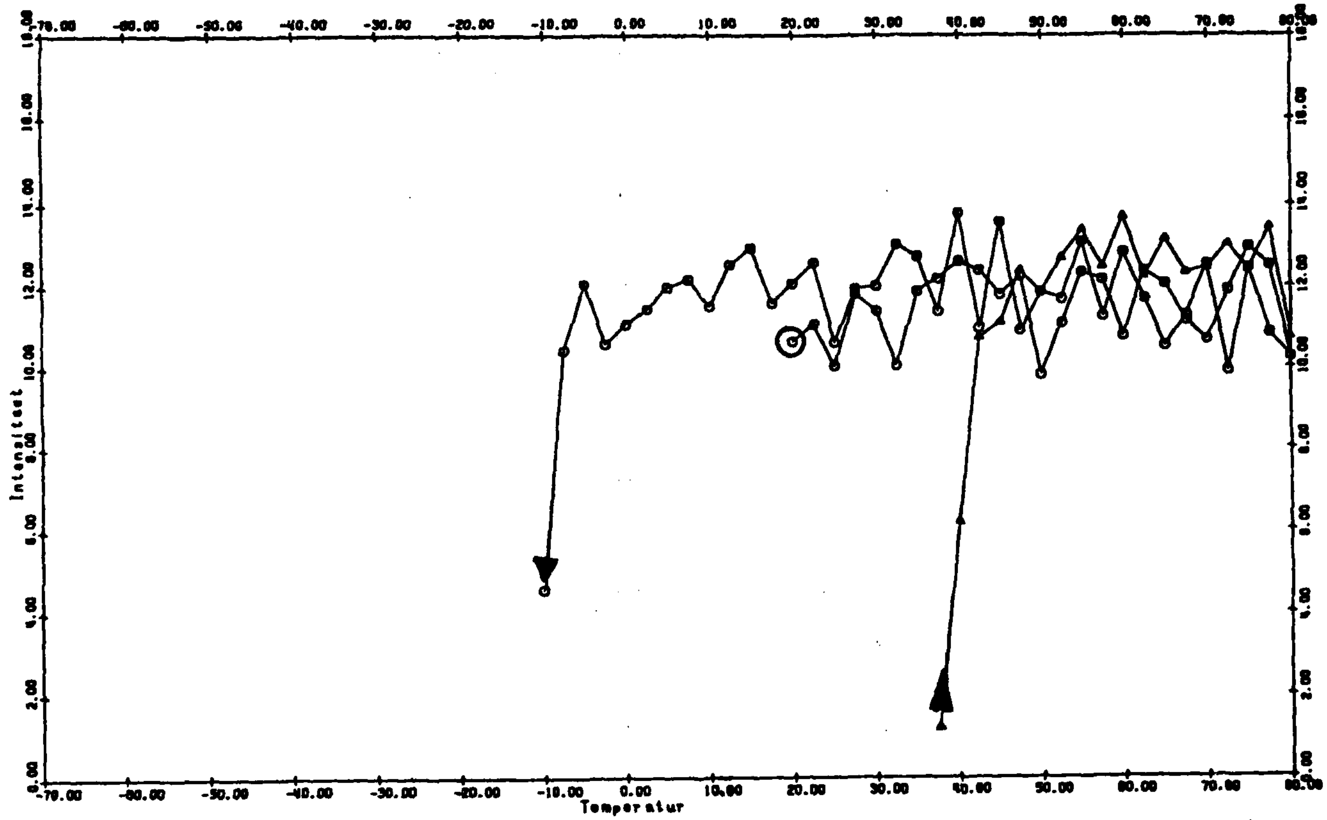
$(220)_{III}$

Intensities



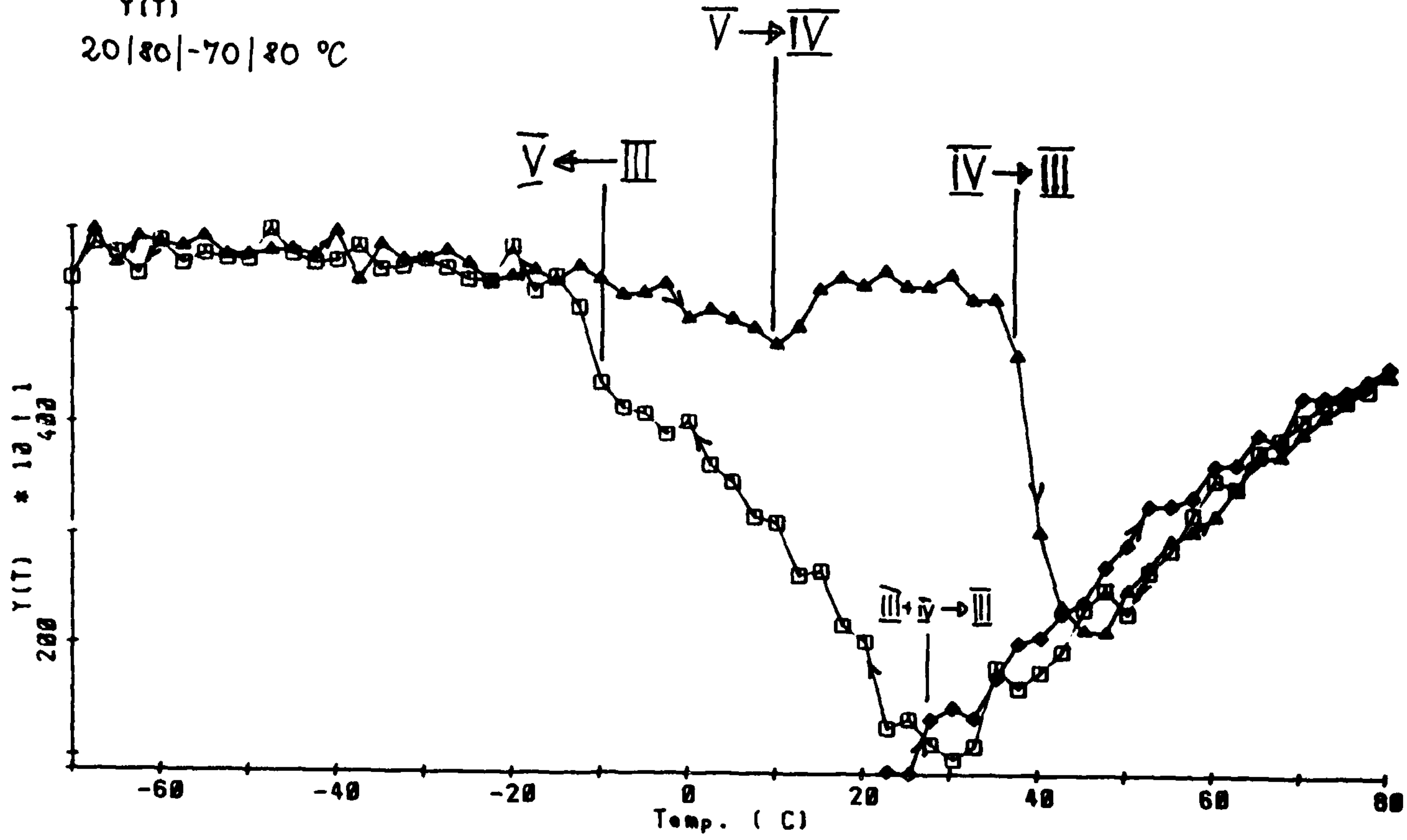
KF 250288
$(220)_{III}$

Intensities



Difference Curve

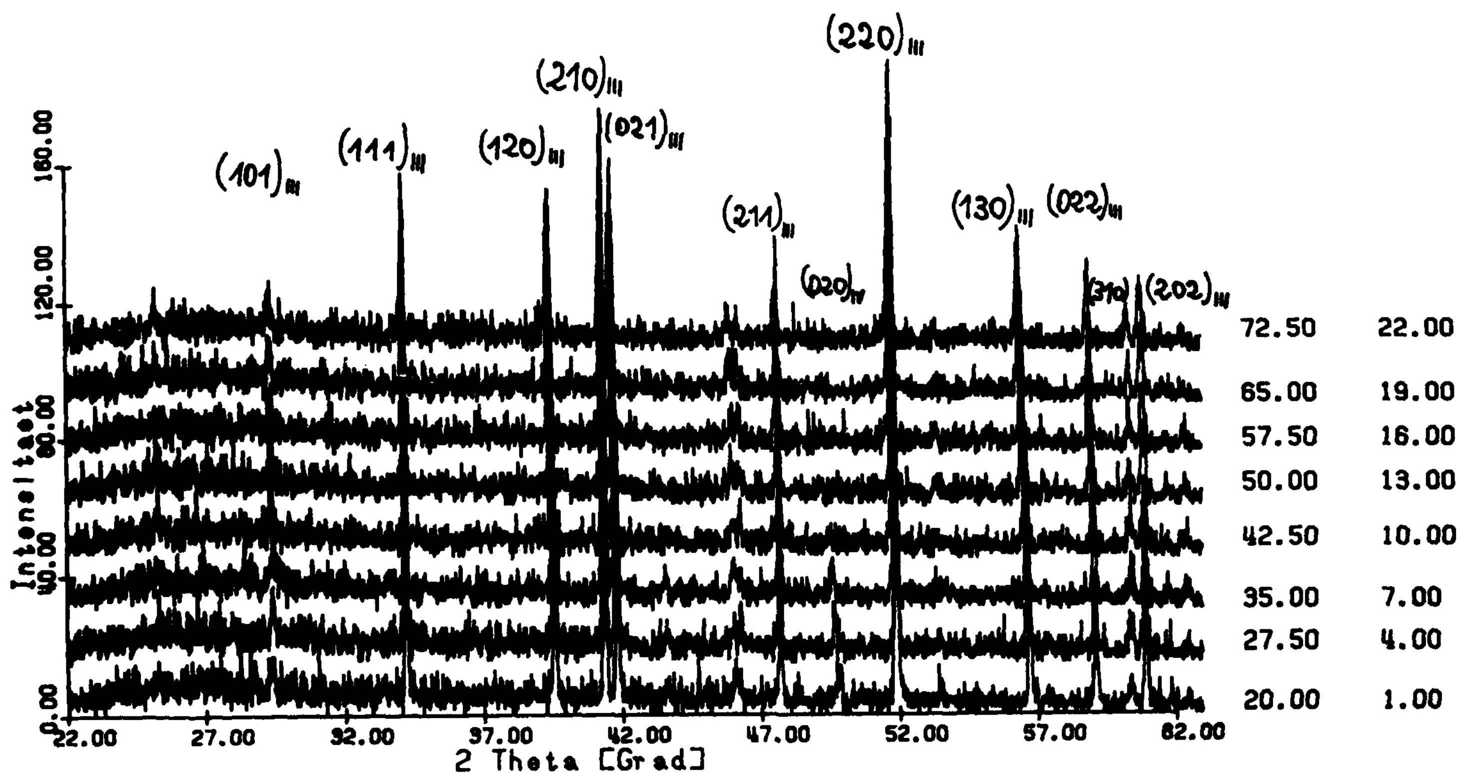
KF250288
Y(T)
20|80|-70|80 °C



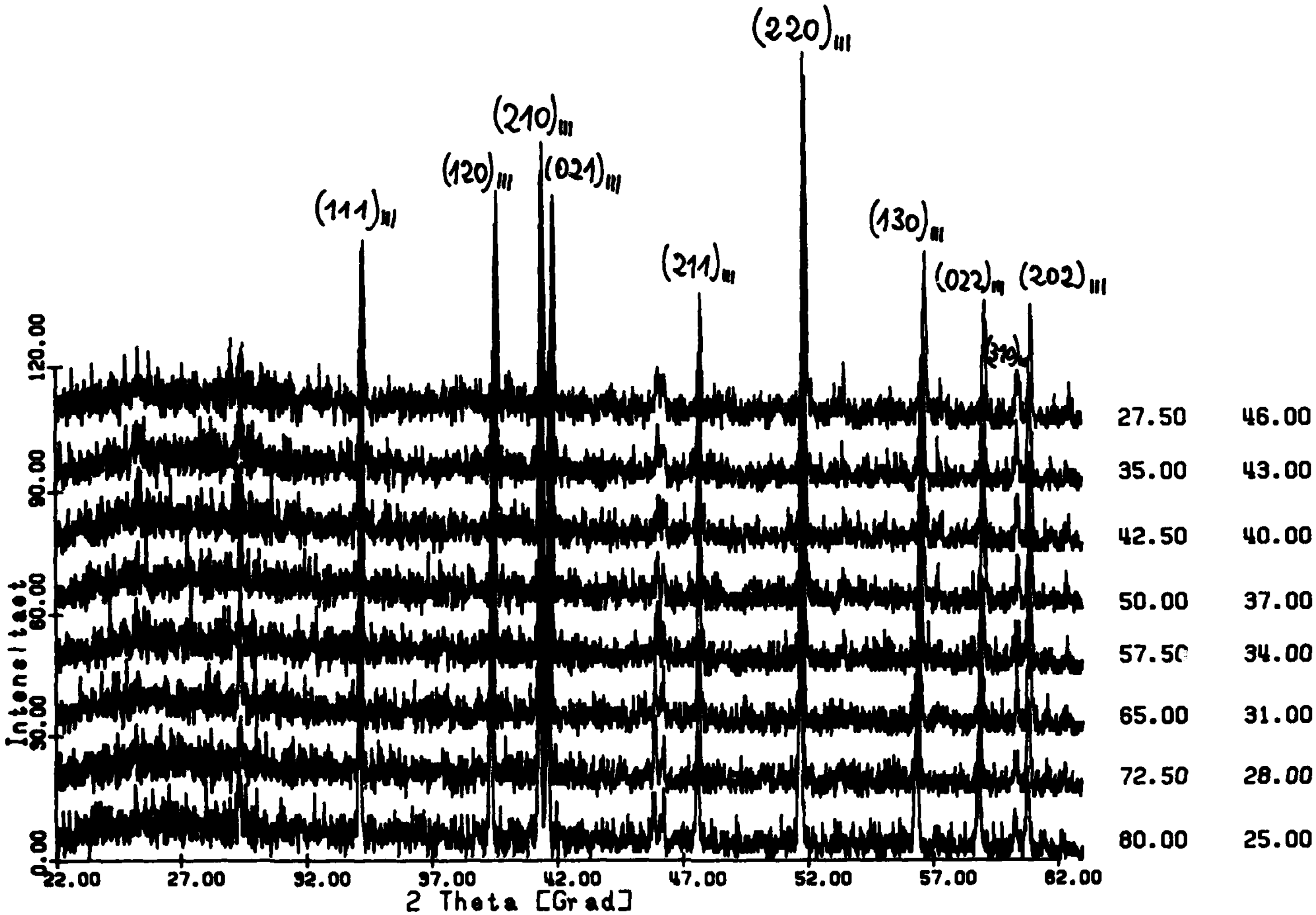
Diffraction Patterns

kf250288

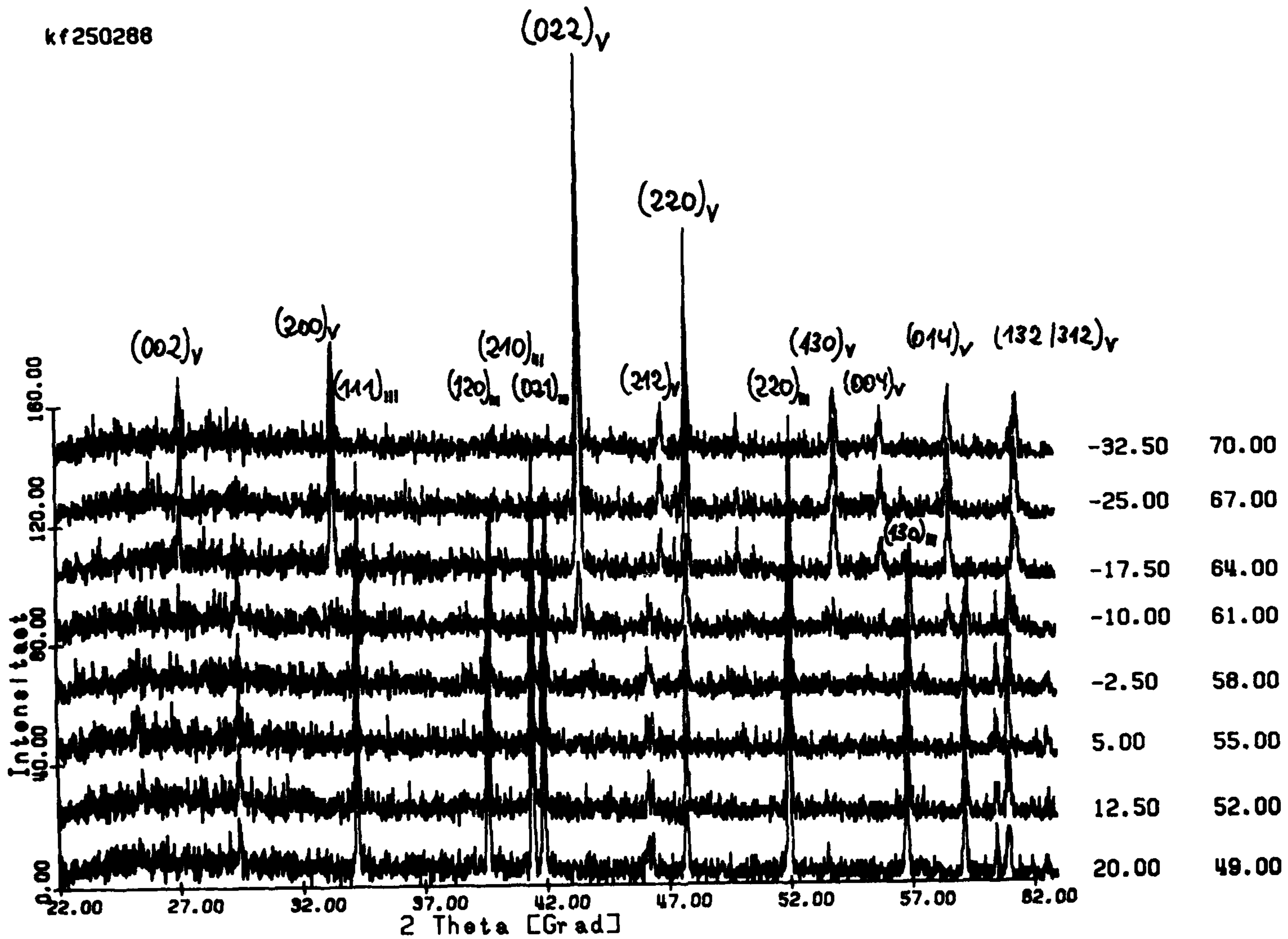
20 | 80 | -70 | 80 °C



kf250288



(111)III
(120)III
(210)III
(021)III
(211)III
(220)III
(130)III
(022)III
(310)III
(202)III
Intensitaet
0.00
30.00
60.00
90.00
120.00
22.00
27.00
32.00
37.00
42.00
47.00
52.00
57.00
62.00
2 Theta [Grad]
27.50 46.00
35.00 43.00
42.50 40.00
50.00 37.00
57.50 34.00
65.00 31.00
72.50 28.00
80.00 25.00


kf250288
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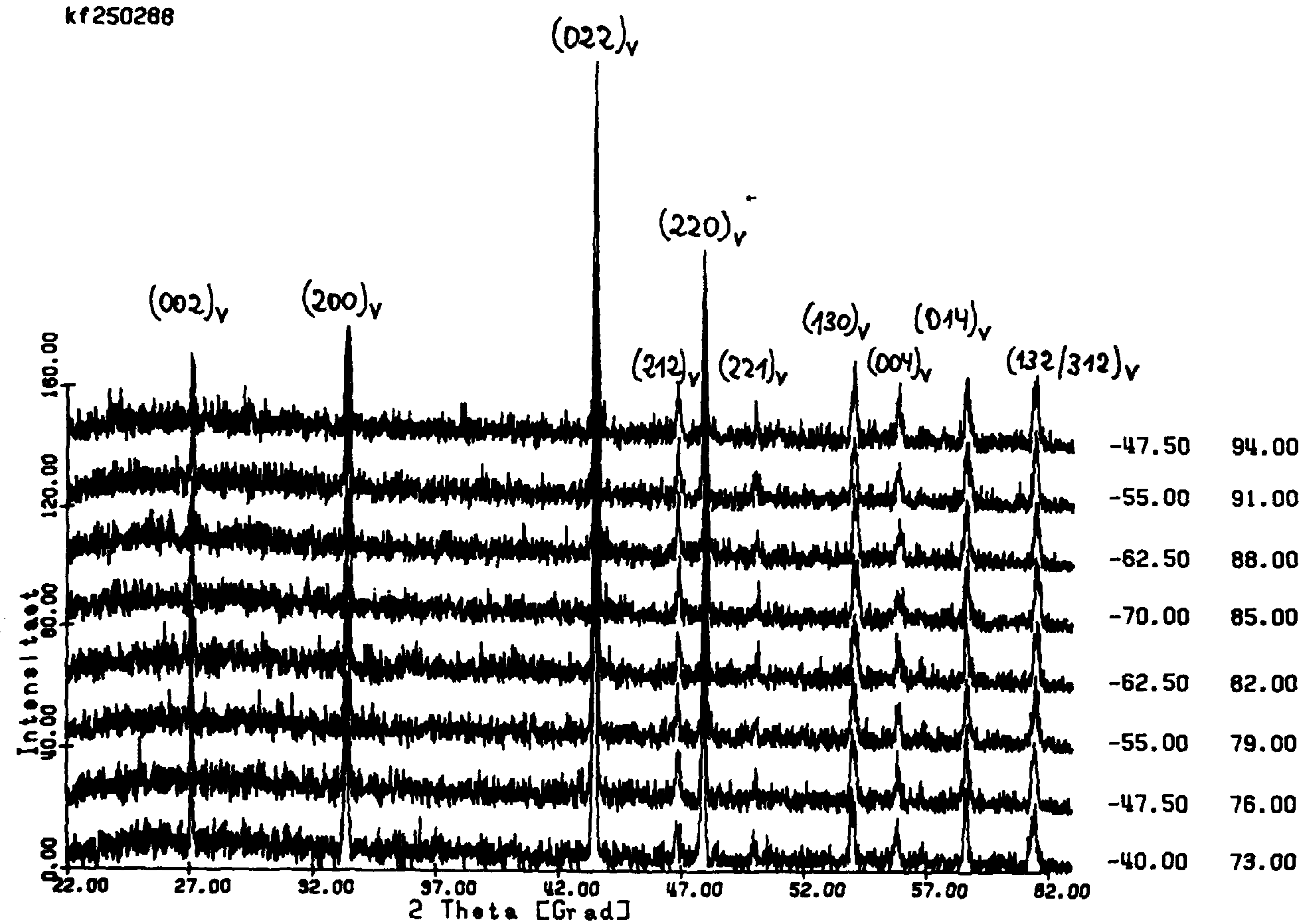
(220)V
(002)V
(200)V
(111)III
(210)III
(120)III
(021)III
(212)V
(220)III
(430)V
(004)V
(014)V
(132/312)V
(130)III
160.00
120.00
80.00
40.00
0.00
Intensitaet
22.00
27.00
32.00
37.00
42.00
47.00
52.00
57.00
82.00
2 Theta [Grad]
-32.50 70.00
-25.00 67.00
-17.50 64.00
-10.00 61.00
-2.50 58.00
5.00 55.00
12.50 52.00
20.00 49.00


kf250288
(002)v
(200)v
(022)v
(220)v
(212)v
(221)v
(130)v
(004)v
(014)v
(132/312)v
Intensitaet
0.00
40.00
80.00
120.00
160.00
22.00
27.00
32.00
37.00
42.00
47.00
52.00
57.00
82.00
2 Theta [Grad]
-47.50 94.00
-55.00 91.00
-62.50 88.00
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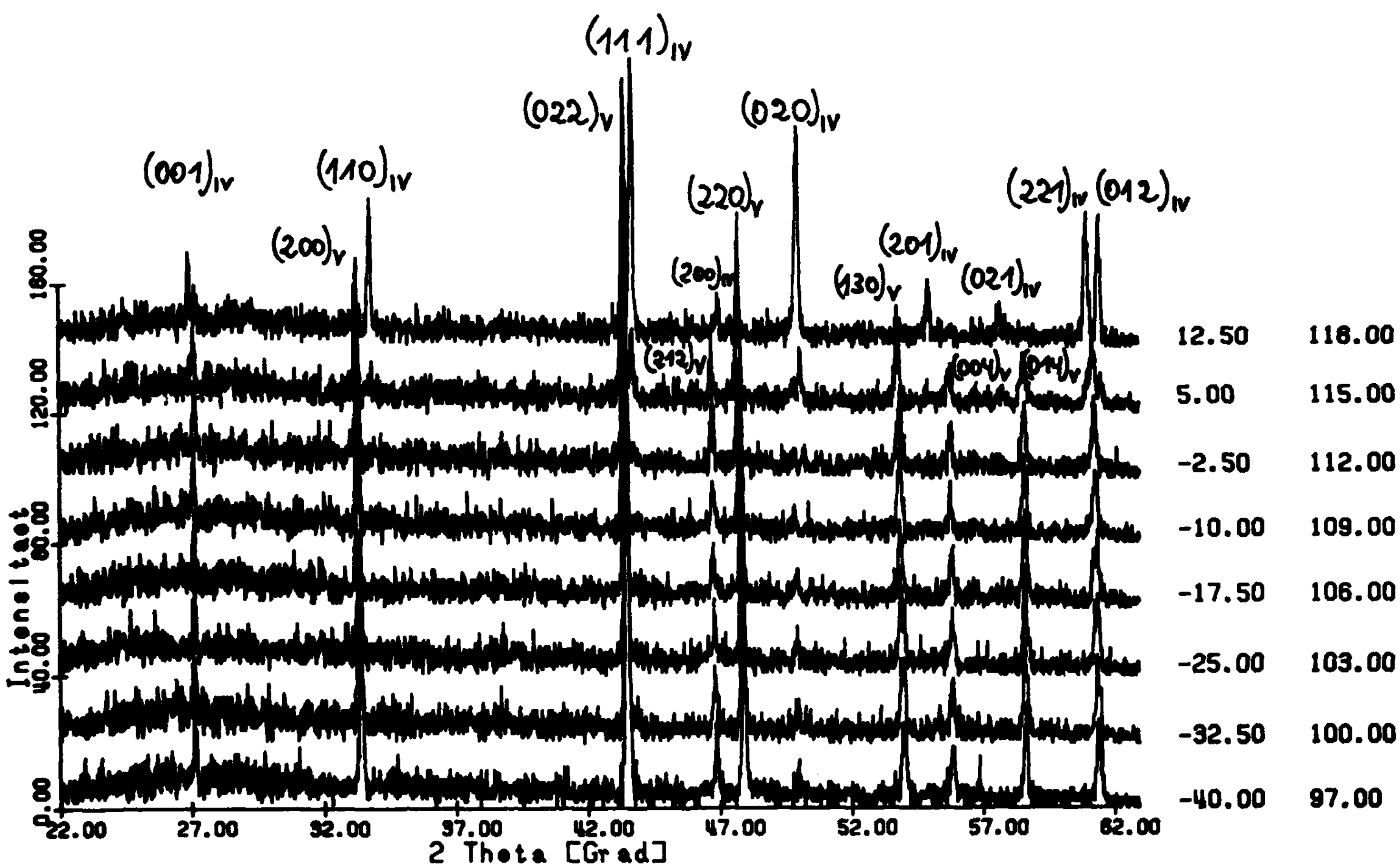
-62.50 82.00
-55.00 79.00
-47.50 76.00
-40.00 73.00

kf250288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(200)V
(110)IV
(022)V
(111)IV
(200)IV
(212)V
(220)V
(020)IV
(130)V
(201)IV
(004)V
(021)IV
(014)V
(221)IV
(012)IV
Intensitaet
0.00
40.00
80.00
120.00
160.00
22.00
27.00
32.00
37.00
42.00
47.00
52.00
57.00
62.00
2 Theta [Grad]
12.50 116.00
5.00 115.00
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-10.00 109.00
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-25.00 103.00
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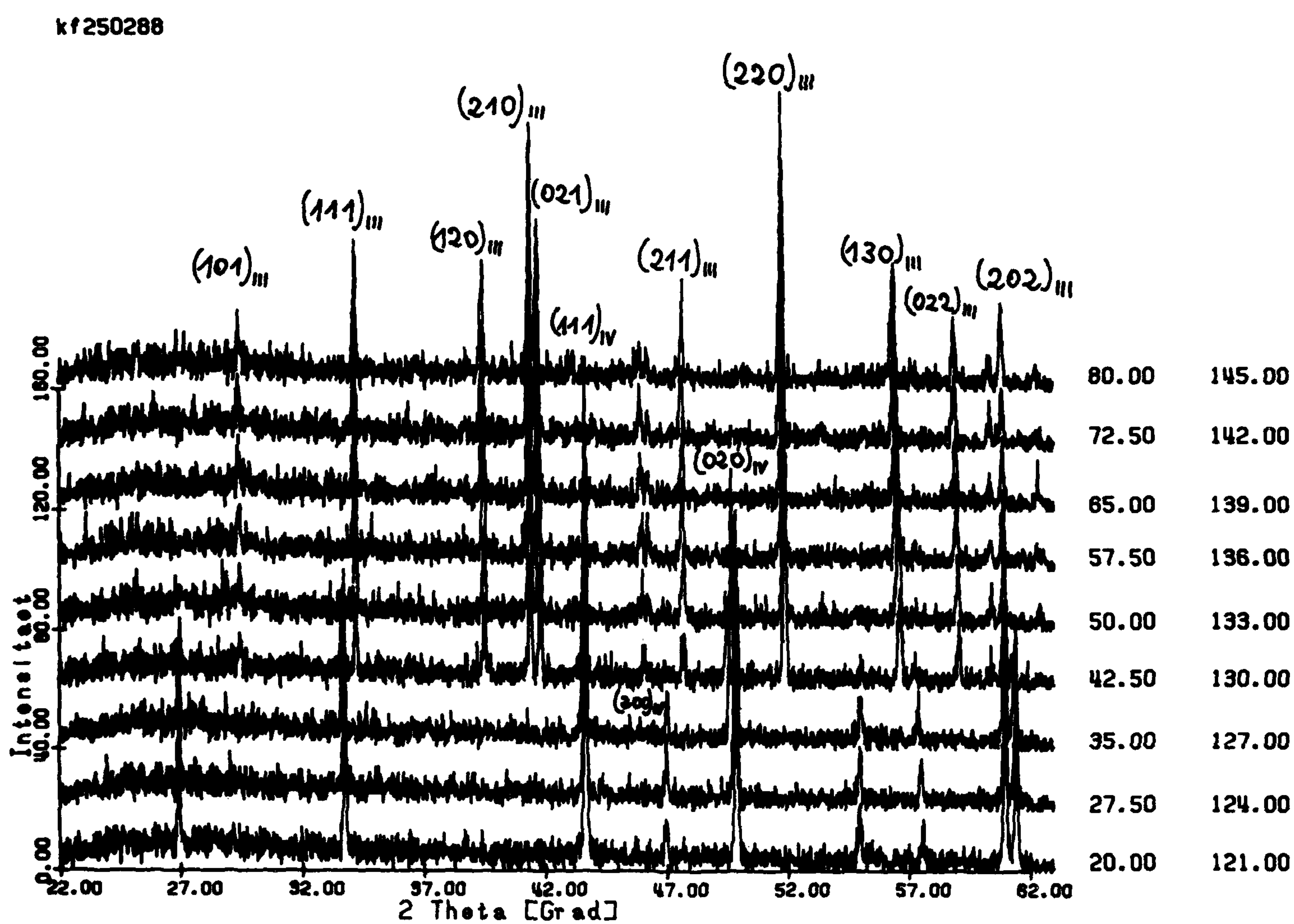
-40.00 97.00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(210)III
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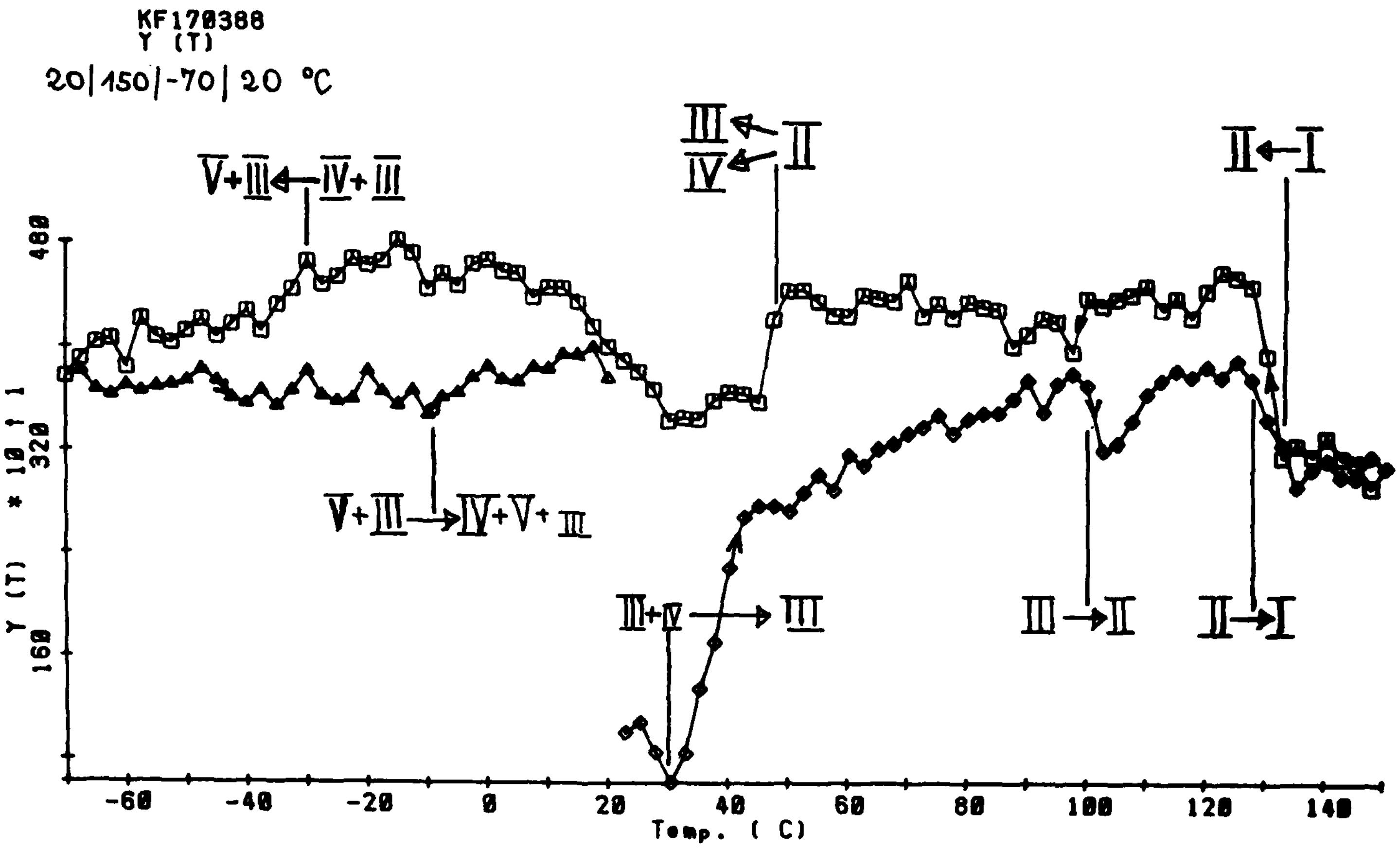
(111)IV
(211)III
(020)IV
(300)IV
(220)III
(130)III
(022)III
(202)III
Intensitaet
2 Theta [Grad]
80.00 145.00
72.50 142.00
65.00 139.00
57.50 136.00
50.00 133.00
42.50 130.00
35.00 127.00
27.50 124.00
20.00 121.00

Series
KF 170388

Temperature
Program
20/150/−70/20

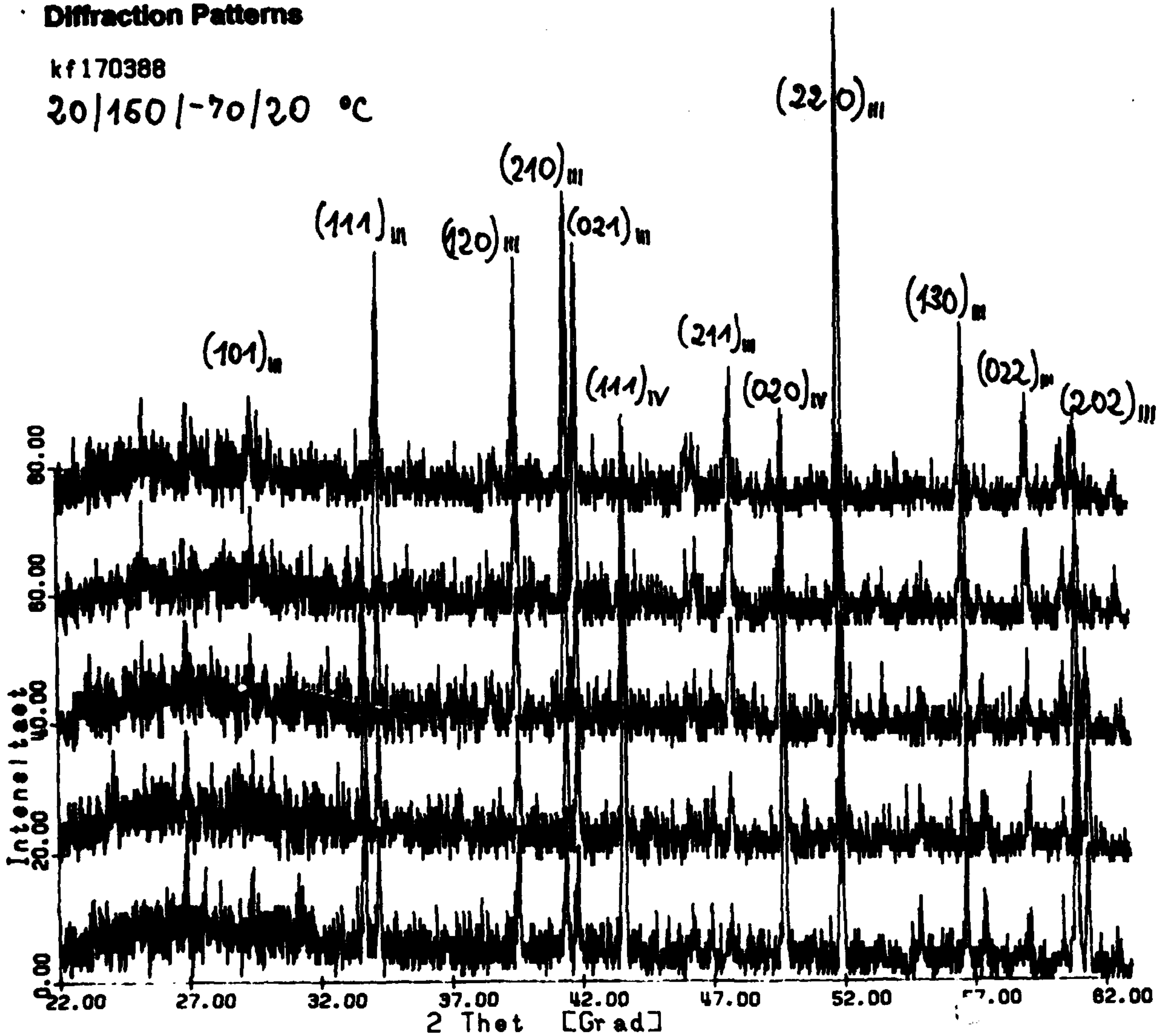
Difference Curve

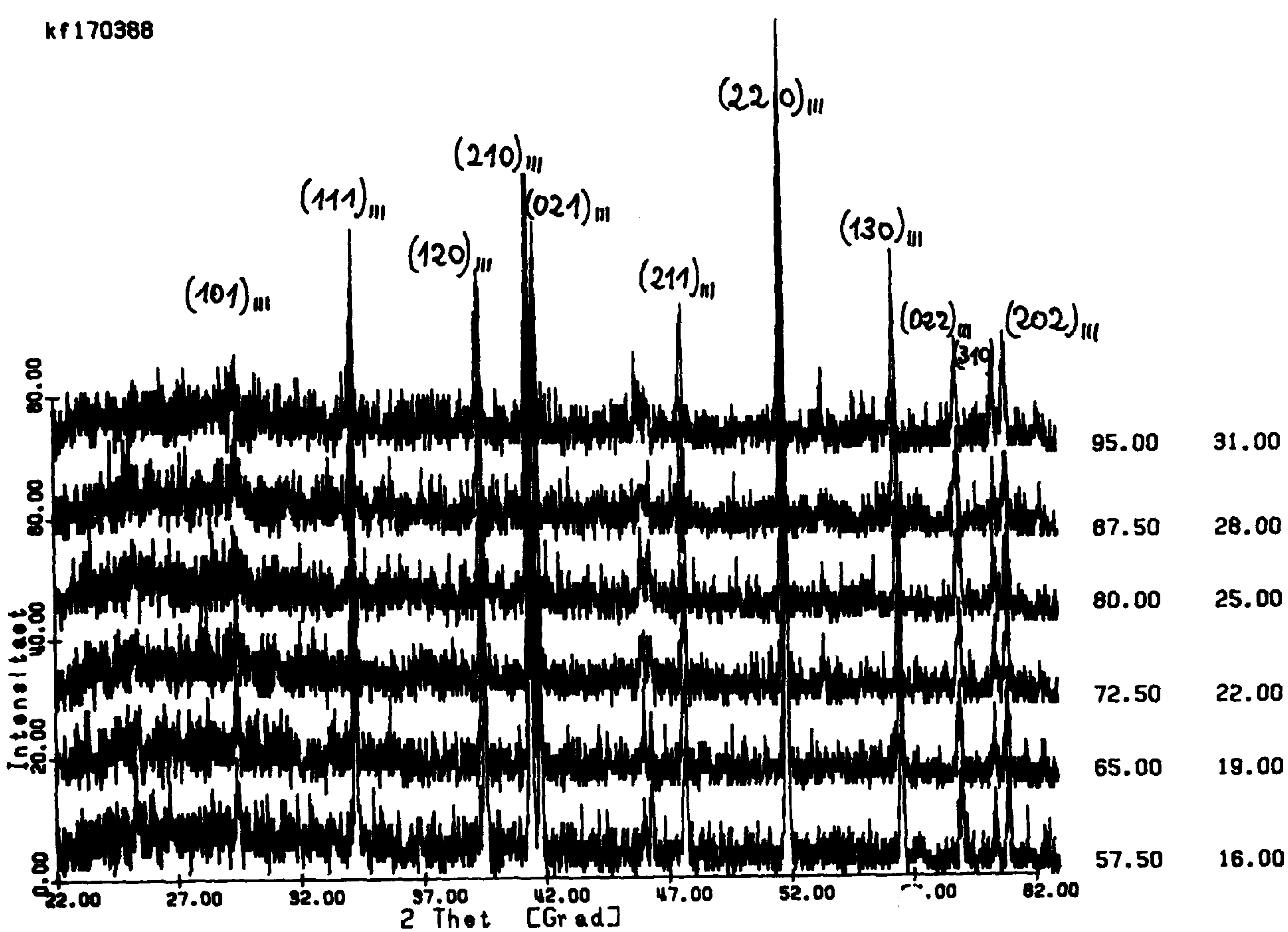


Diffraction Patterns

kf170388

20/150/-70/20 °C




kf170368
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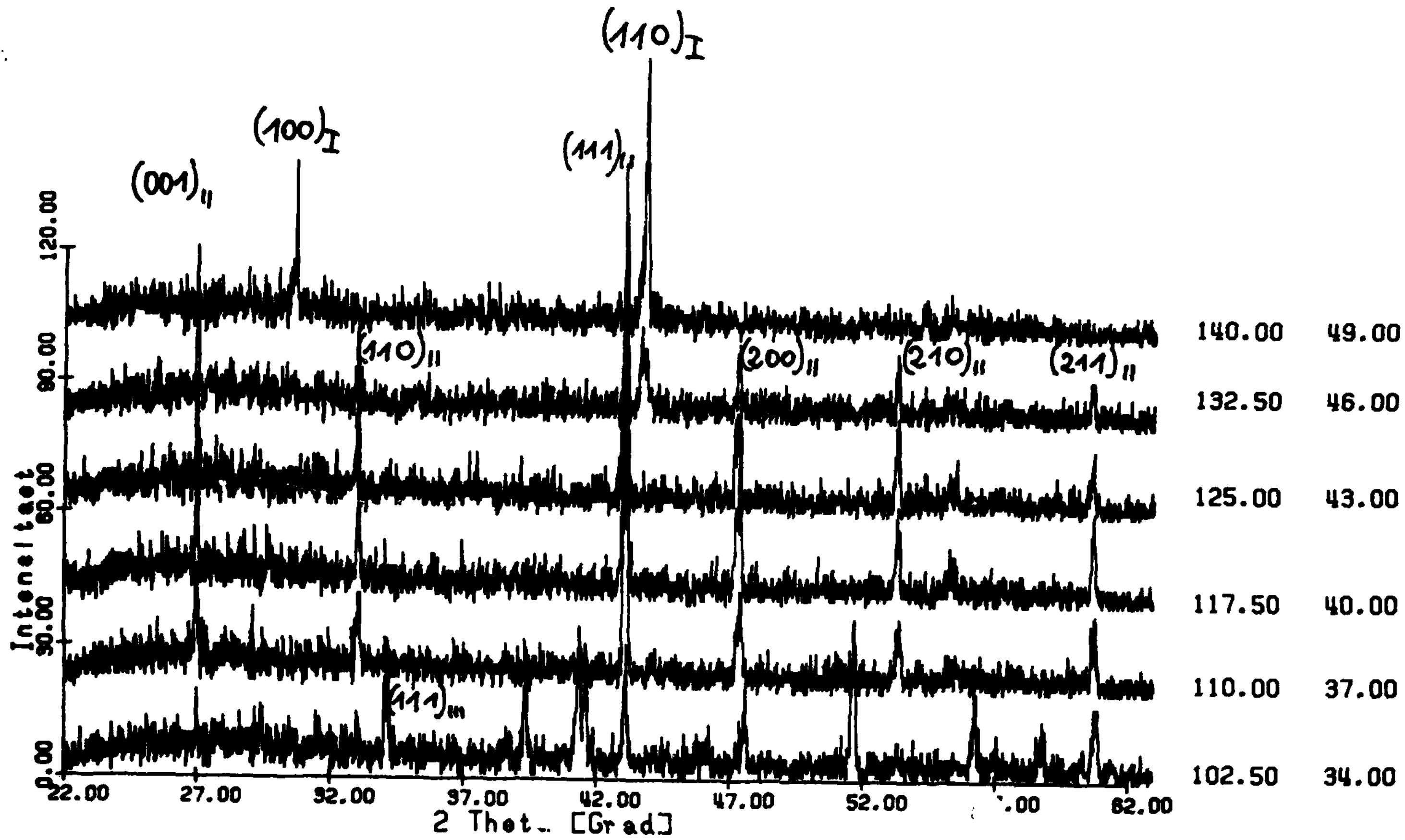
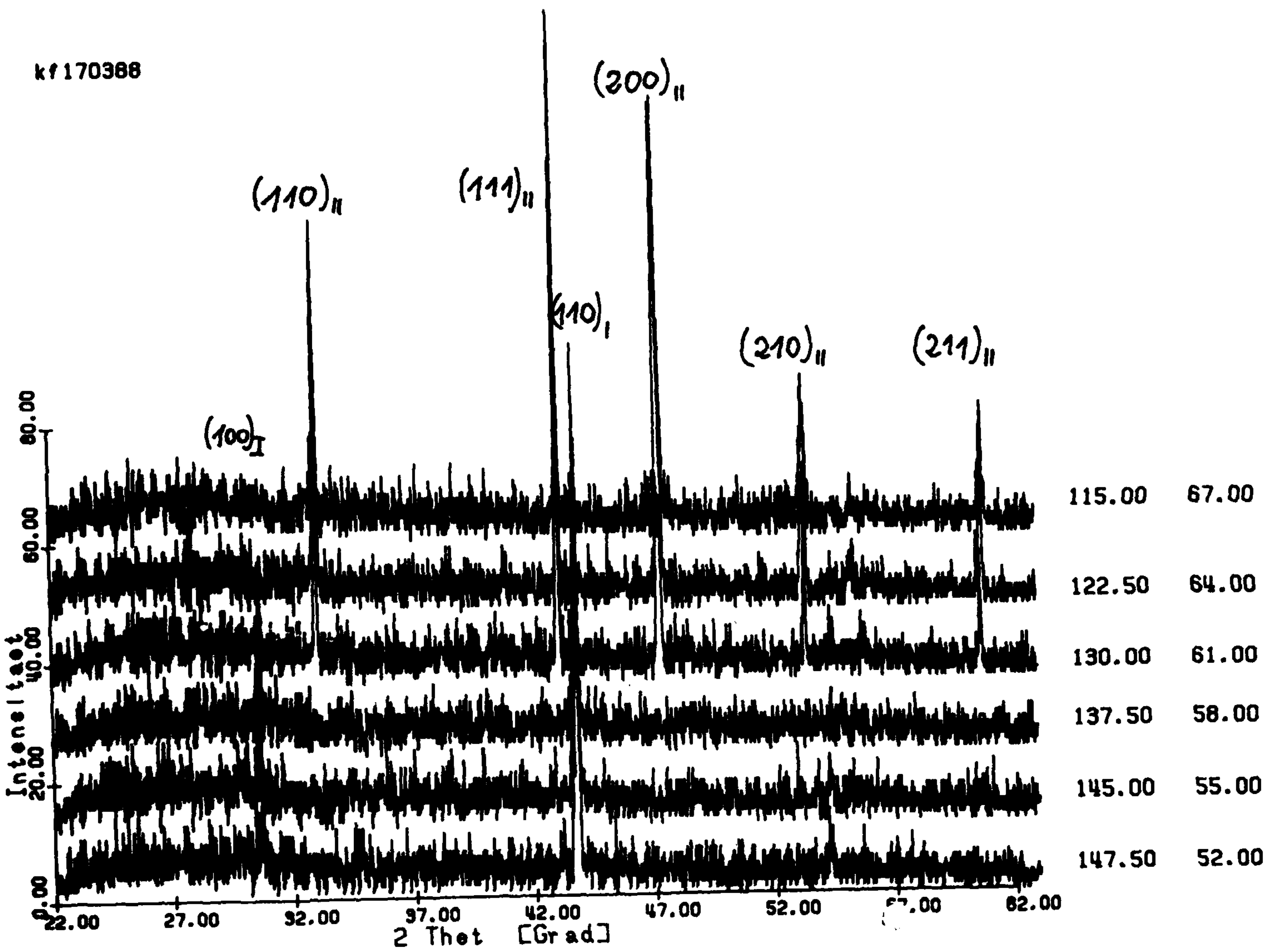
(120)III
(210)III
(021)III
(211)III
(220)III
(130)III
(022)III
(310)
(202)III
95.00 31.00
87.50 28.00
80.00 25.00
72.50 22.00
65.00 19.00
57.50 16.00
Intensitaet
2 Thet [Grad]

kf170388

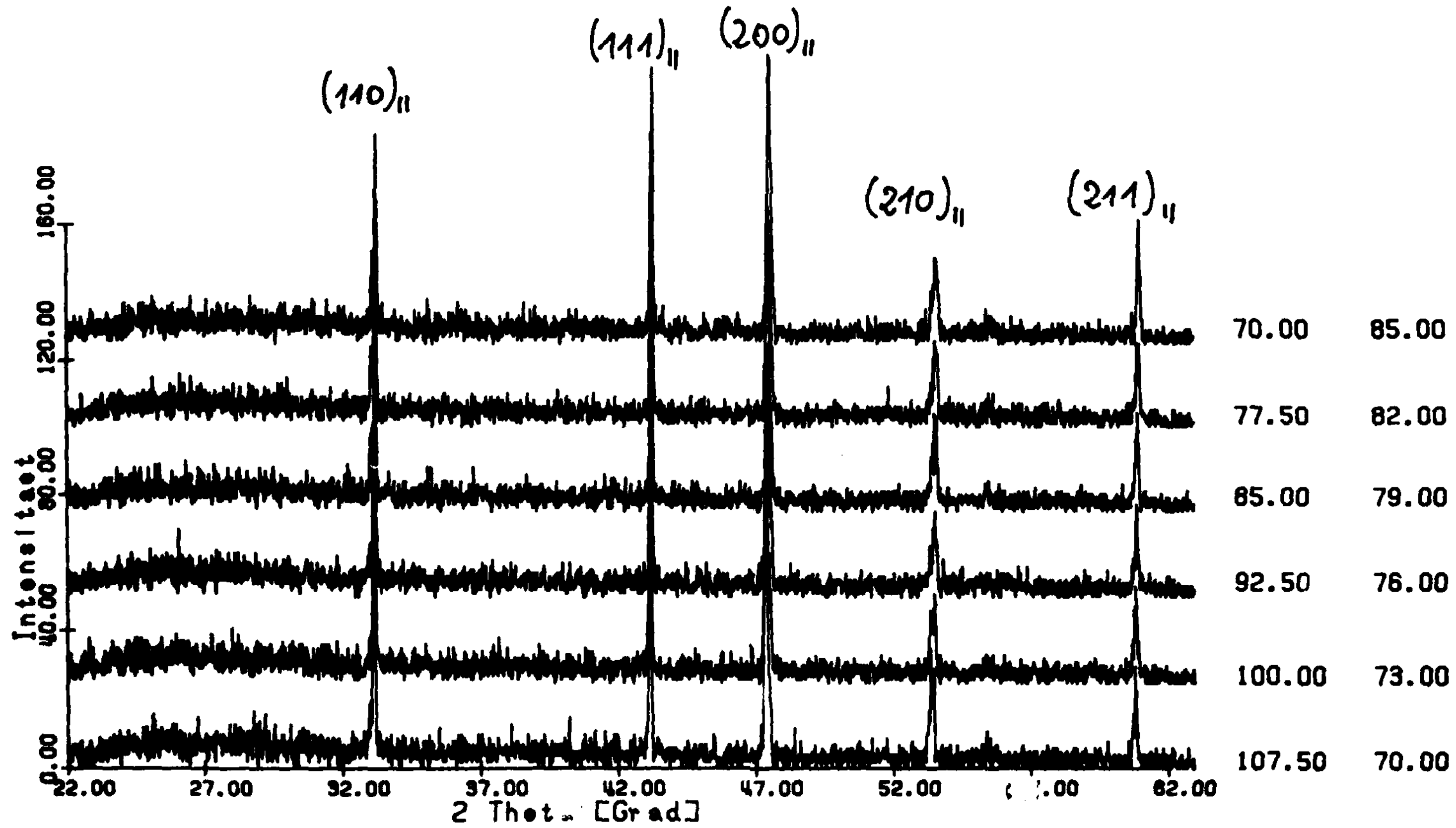


(001)II
(100)I
(110)I
(111)II
(110)II
(200)II
(210)II
(211)II
(111)III
Intensitaet
0.00
30.00
60.00
90.00
120.00
22.00
27.00
32.00
37.00
42.00
47.00
52.00
.00
62.00
2 Thet.. [Grad]
140.00 49.00
132.50 46.00
125.00 43.00
117.50 40.00
110.00 37.00
102.50 34.00



kf170388
(110)II
(111)II
(200)II
(110)I
(210)II
(211)II
(100)I
80.00
60.00
40.00
20.00
0.00
Intensitaet
22.00
27.00
32.00
37.00
42.00
47.00
52.00
62.00
2 Thet [Grad]
115.00 67.00
122.50 64.00
130.00 61.00
137.50 58.00
145.00 55.00
147.50 52.00

kf170388



(110)II
(111)II
(200)II
(210)II
(211)II
Intensitaet
0.00
40.00
80.00
120.00
160.00
22.00
27.00
32.00
37.00
42.00
47.00
52.00
62.00
2 Theta [Grad]
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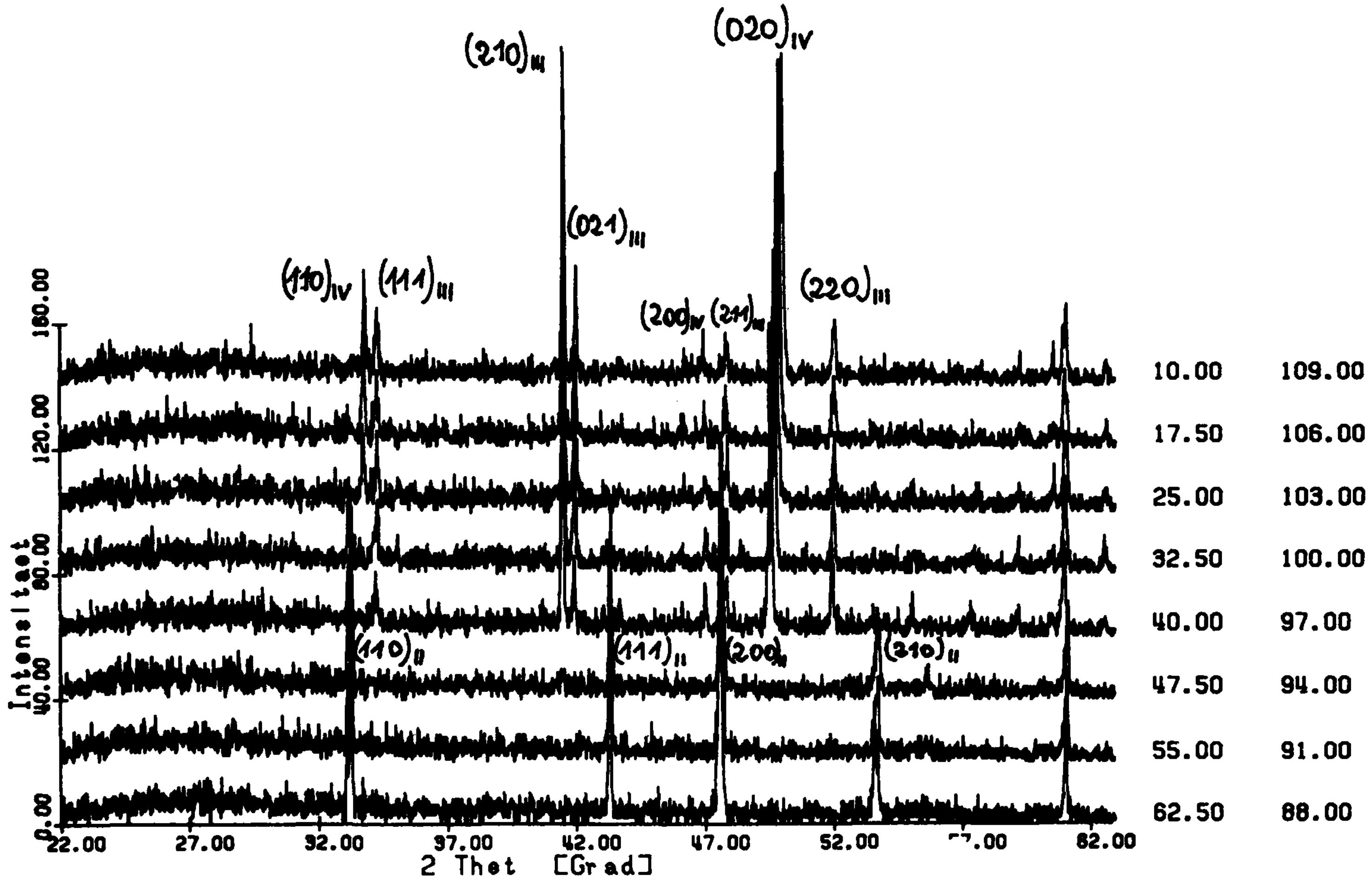
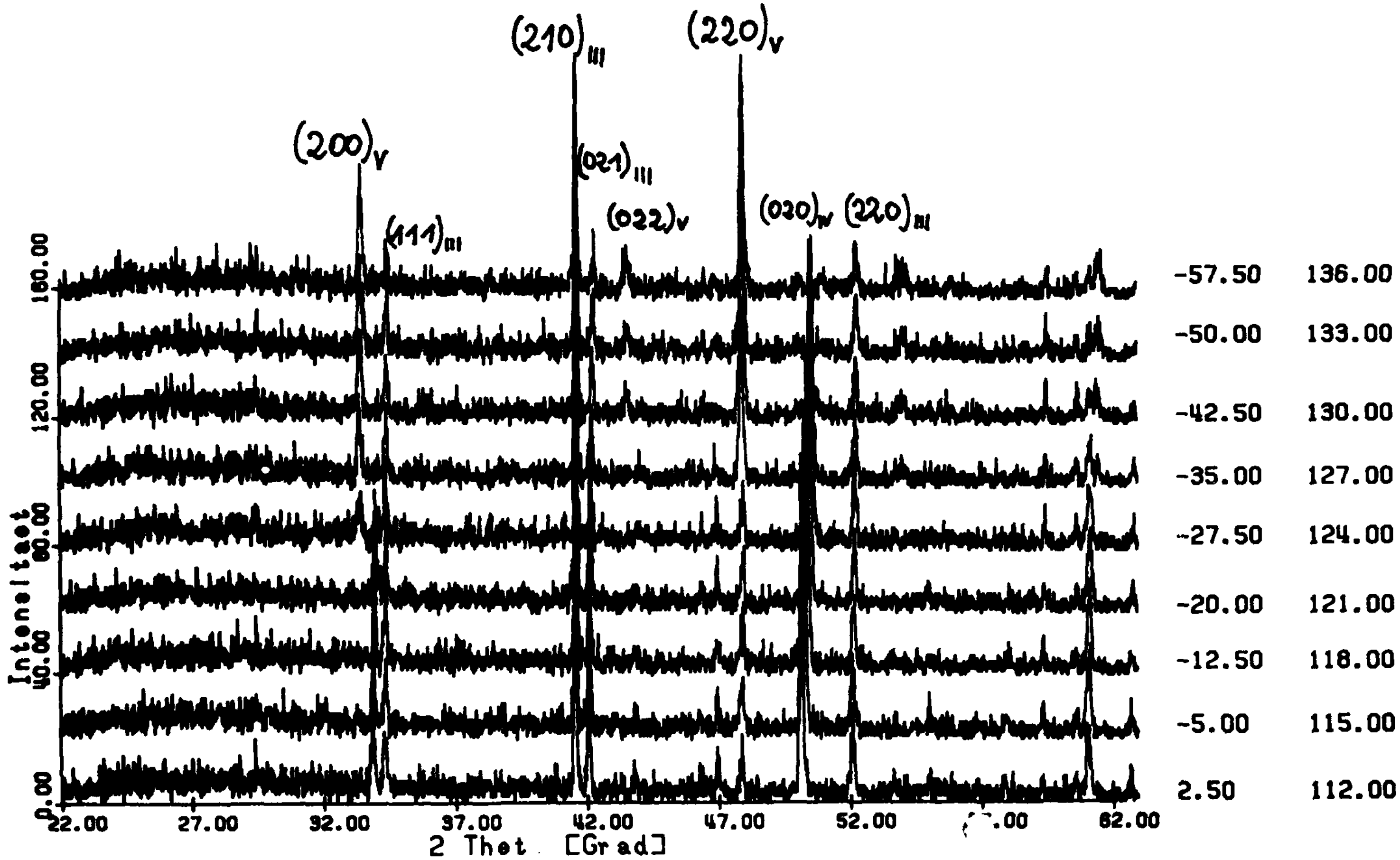
77.50 82.00
85.00 79.00
92.50 76.00
100.00 73.00
107.50 70.00

kf170388

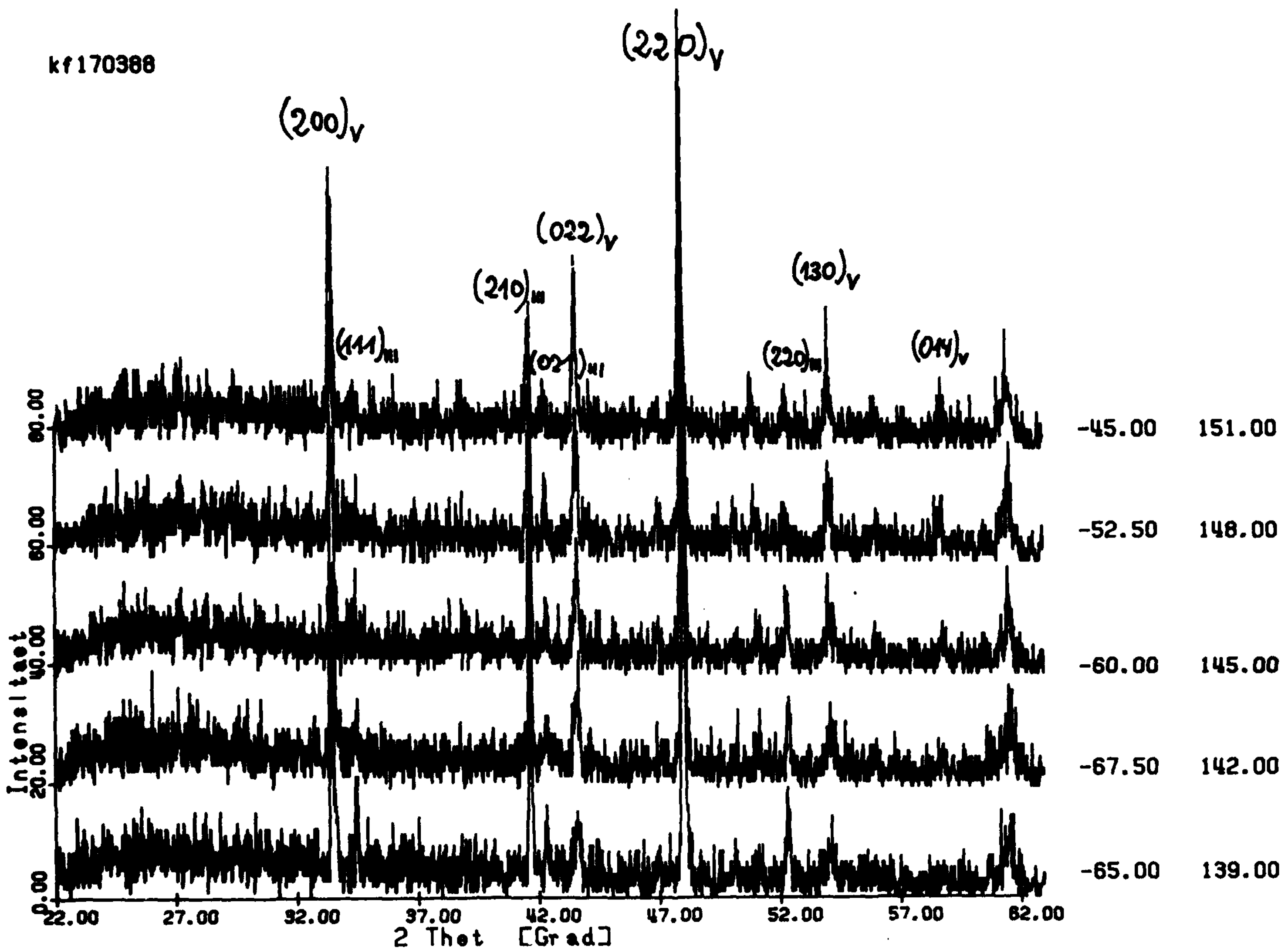


(210)III
(020)IV
(021)III
(110)IV
(111)III
(200)IV
(211)III
(220)III
(110)II
(111)II
(200)II
(310)II
160.00
120.00
80.00
40.00
0.00
Intensitaet
22.00
27.00
32.00
37.00
42.00
47.00
52.00
57.00
62.00
2 Thet [Grad]
10.00 109.00
17.50 106.00
25.00 103.00
32.50 100.00
40.00 97.00
47.50 94.00
55.00 91.00
62.50 88.00

kf170388



(200)v
(111)III
(210)III
(021)III
(022)v
(220)v
(020)v
(220)III
Intensitaet
0.00
40.00
80.00
120.00
160.00
22.00
27.00
32.00
37.00
42.00
47.00
52.00
62.00
2 Thet [Grad]
-57.50 136.00
-50.00 133.00
-42.50 130.00
-35.00 127.00
-27.50 124.00
-20.00 121.00
-12.50 118.00
-5.00 115.00
2.50 112.00


kf170388
(200)V
(111)M
(210)M
(021)M
(022)V
(220)V
(220)M
(130)V
(014)V
Intensitaet
0.00
20.00
40.00
60.00
80.00
22.00
27.00
32.00
37.00
42.00
47.00
52.00
57.00
62.00
2 Thet [Grad]
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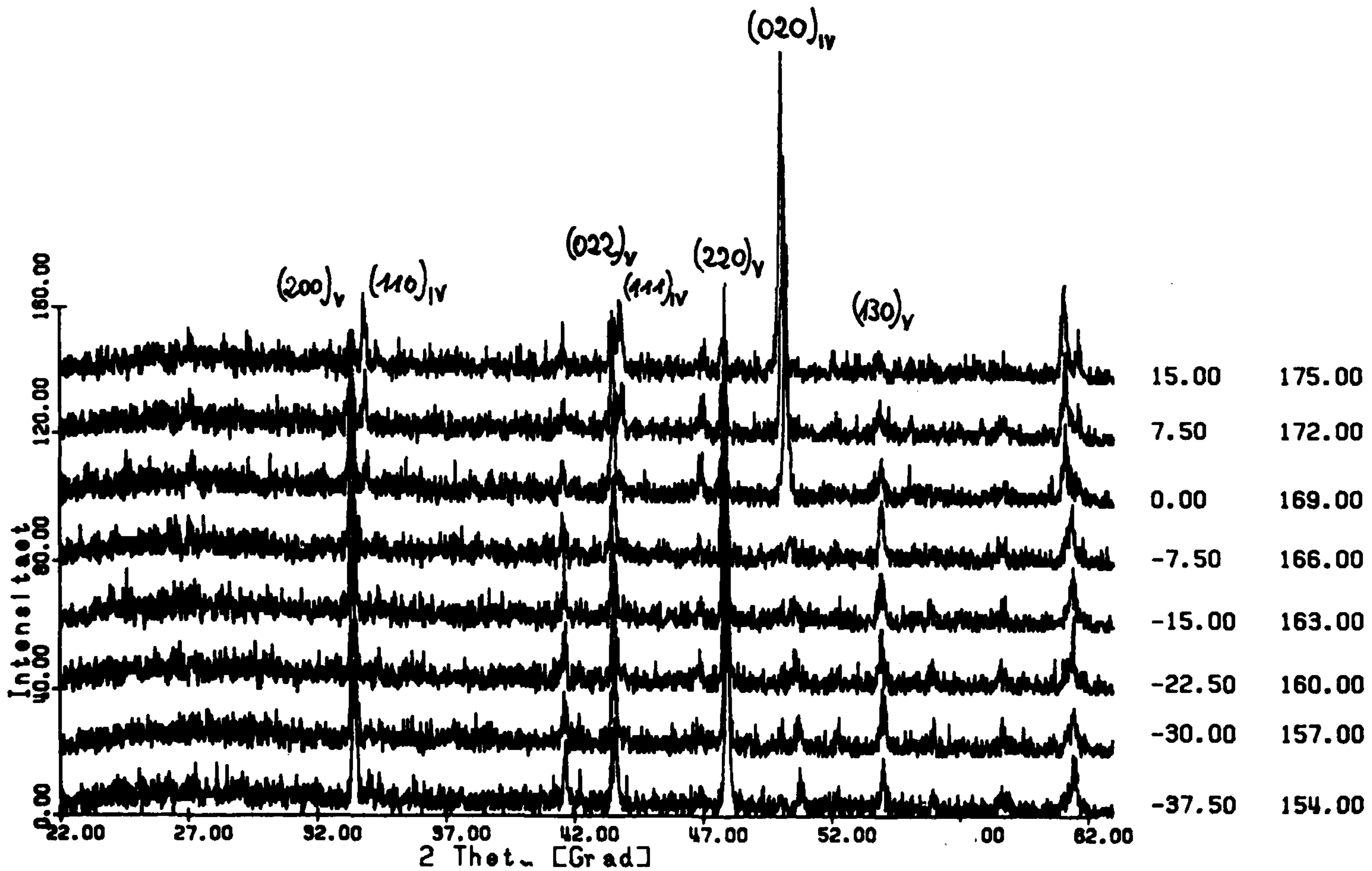
-67.50 142.00
-65.00 139.00

kf170388



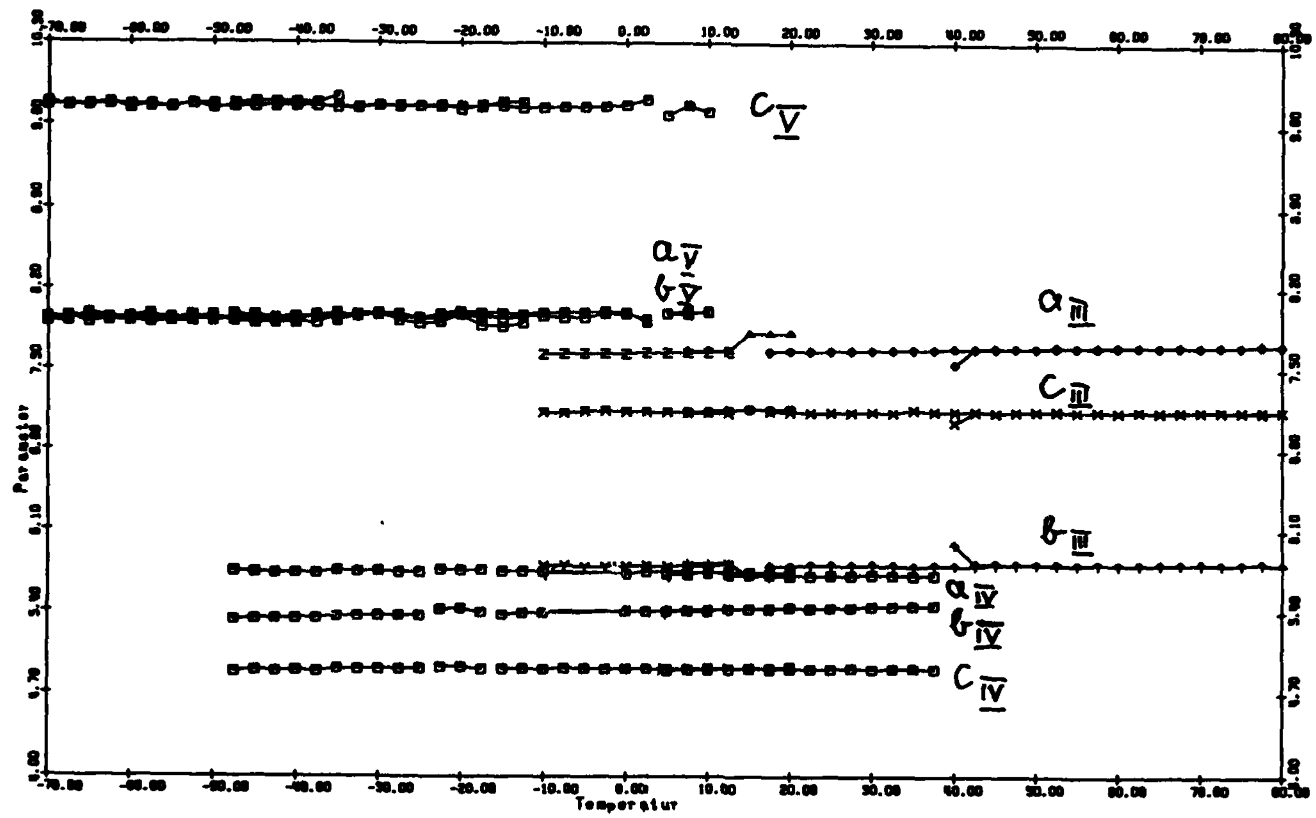
(020)IV
(200)V
(110)IV
(022)V
(111)IV
(220)V
(130)V
Intensitaet
0.00
40.00
80.00
120.00
180.00
22.00
27.00
32.00
37.00
42.00
47.00
52.00
.00
62.00
2 Thet~ [Grad]
15.00 175.00
7.50 172.00
0.00 169.00
-7.50 166.00
-15.00 163.00
-22.50 160.00
-30.00 157.00
-37.50 154.00

Series
KF 230388

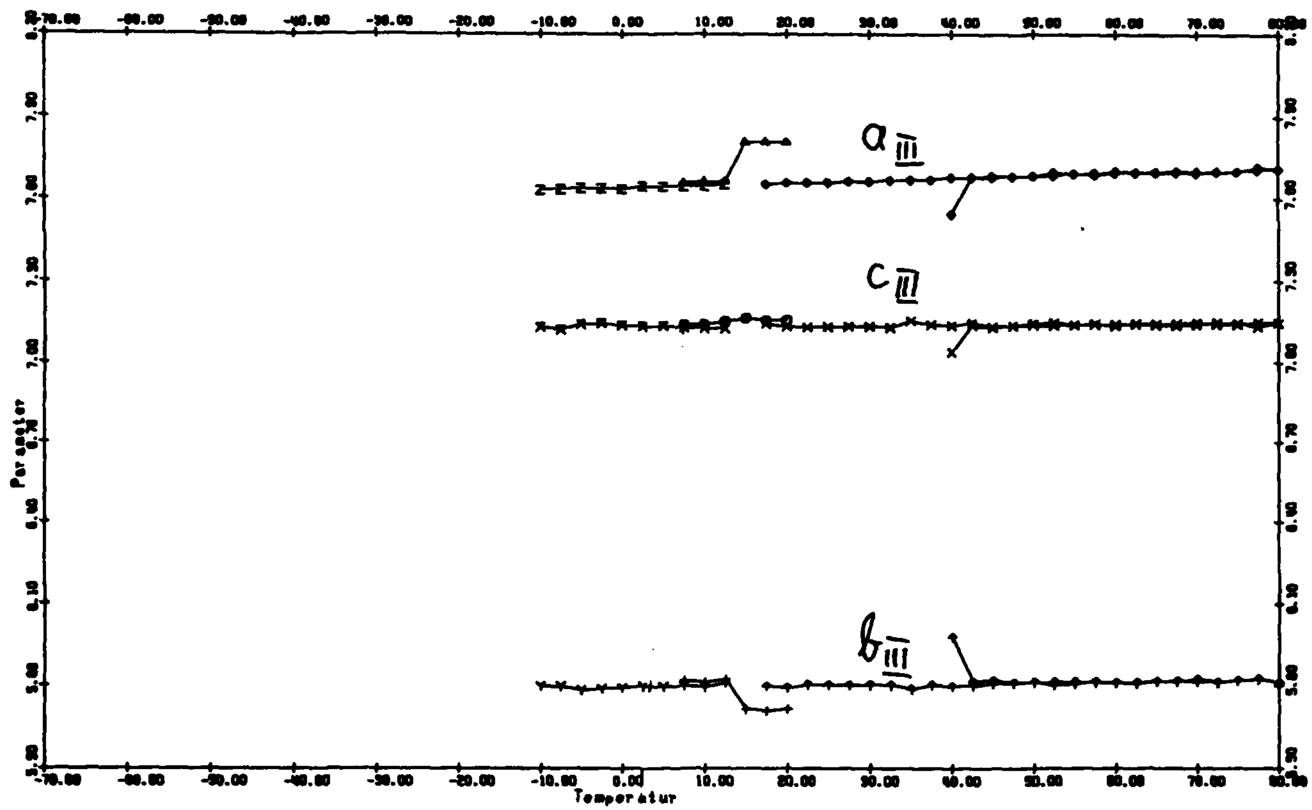
Temperature
Program
20/−70/80/−70

kF 230388
20|-70|80|-70

Lattice Parameters

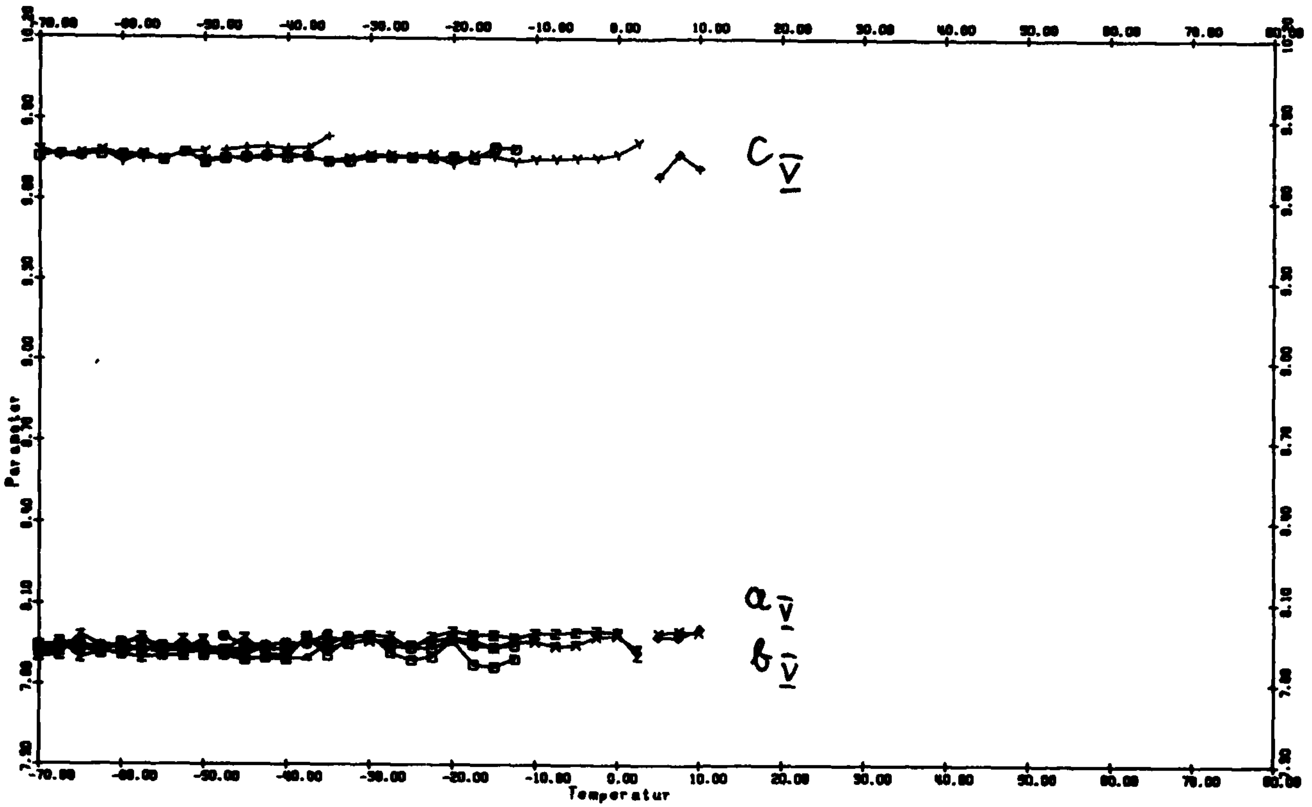


KF 230388
20/-70/80/-70

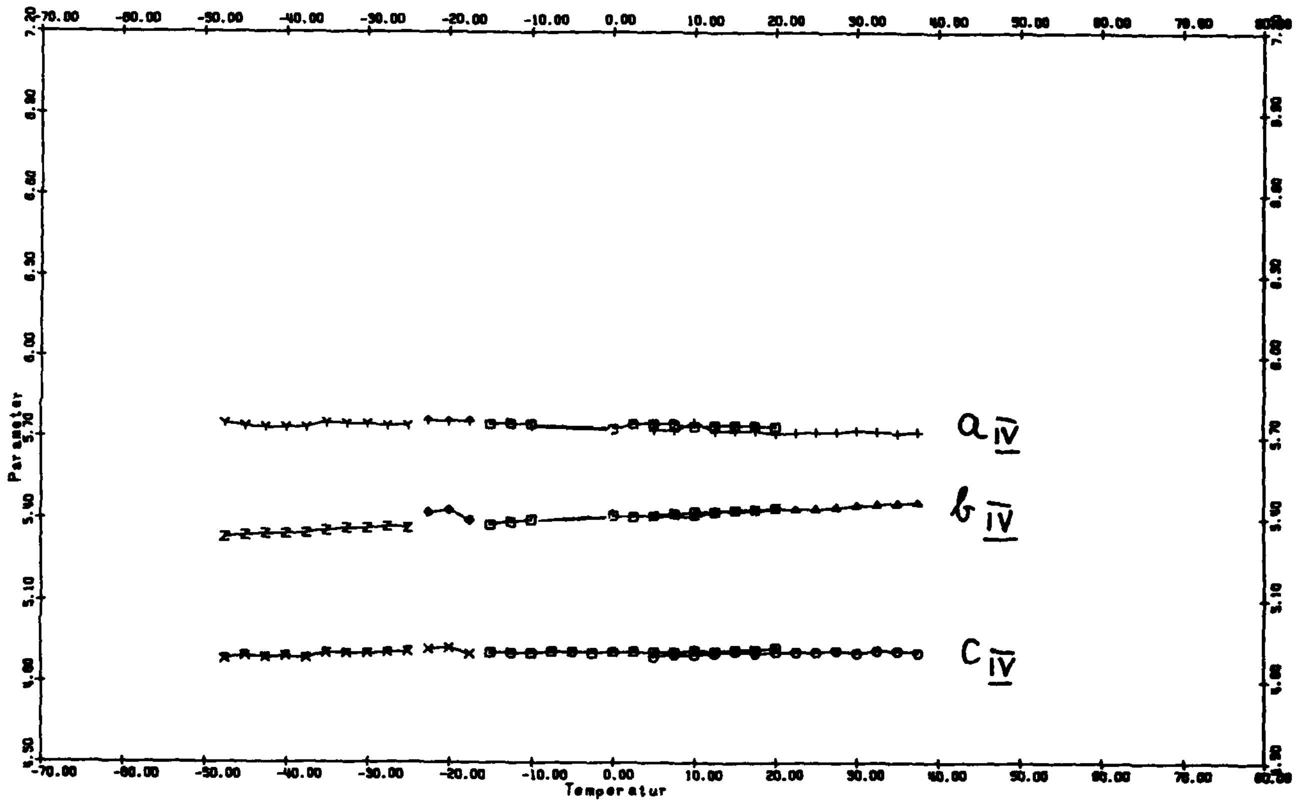


KF 230388
20|-70|80|-70

Lattice Parameters

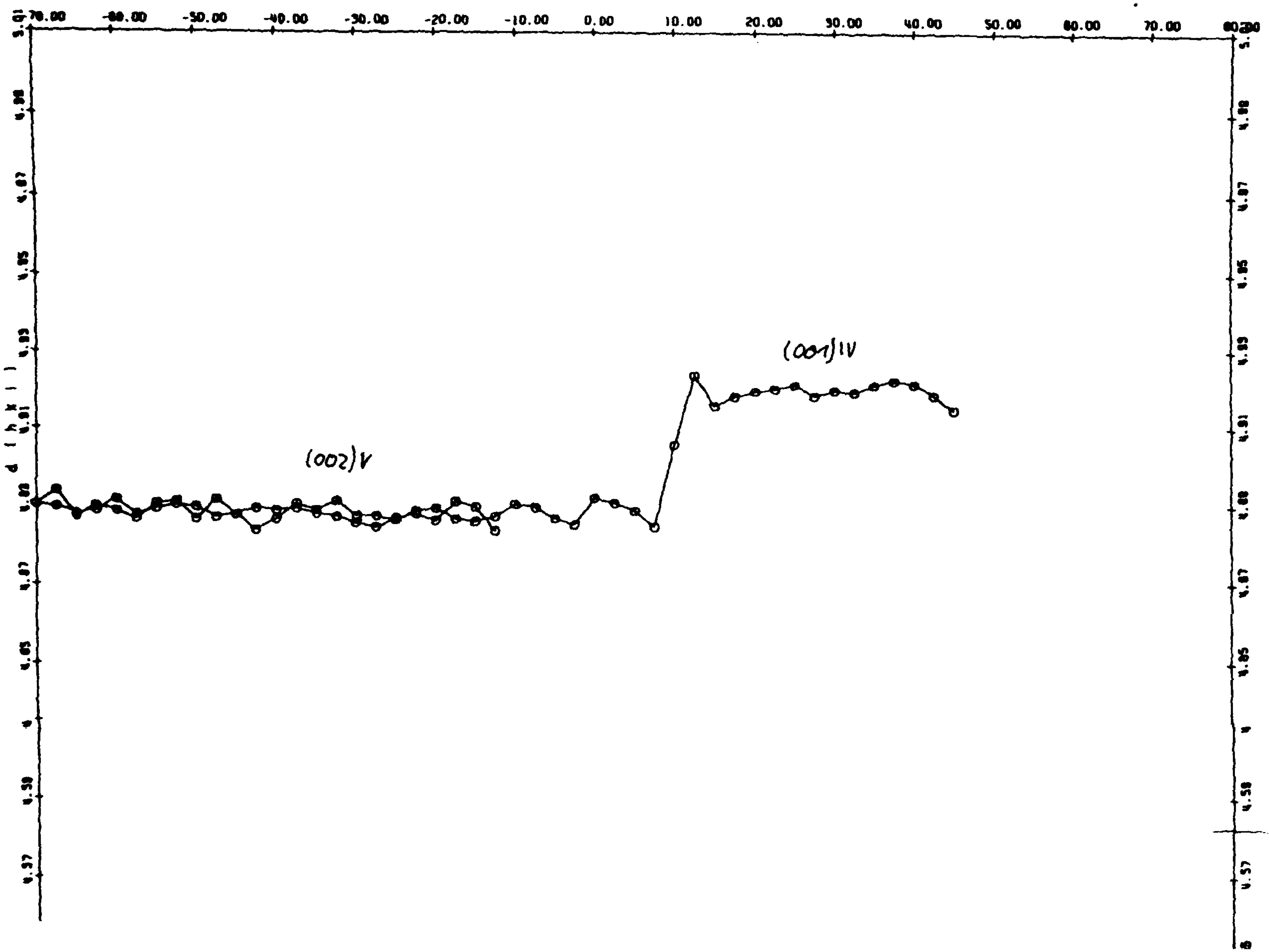


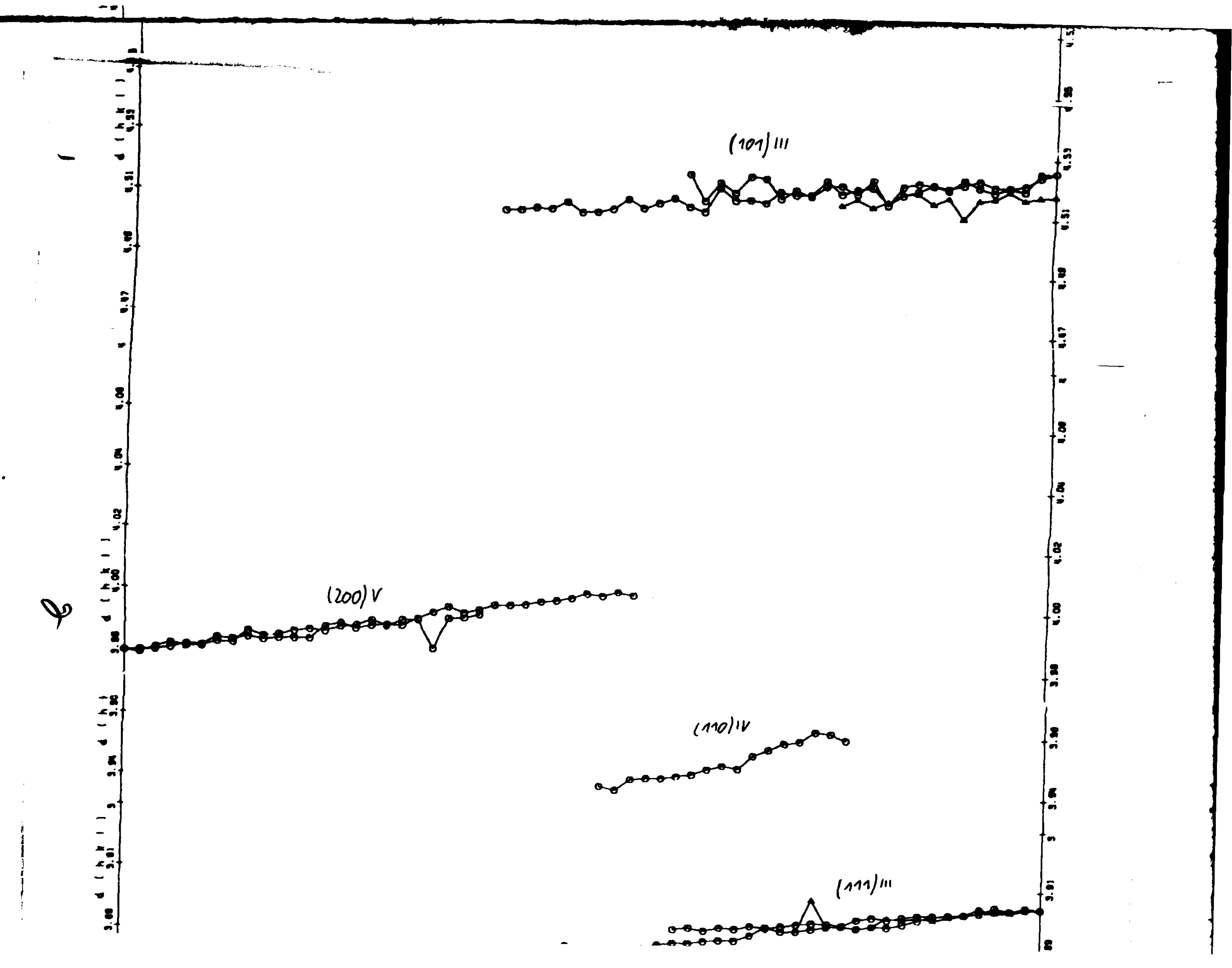
KF 230388
201-70/801-70



K7250288

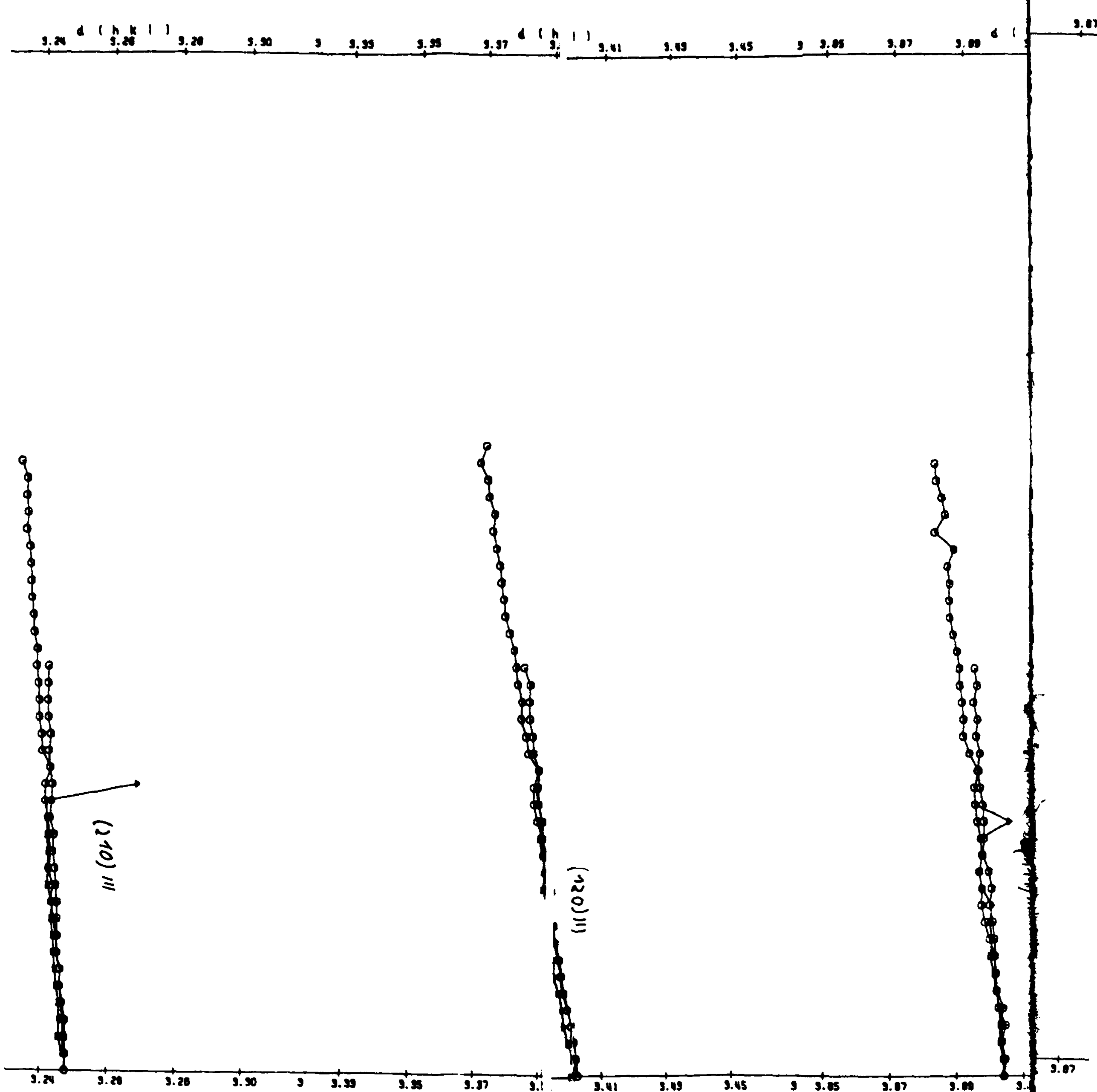
Lattice Distances (

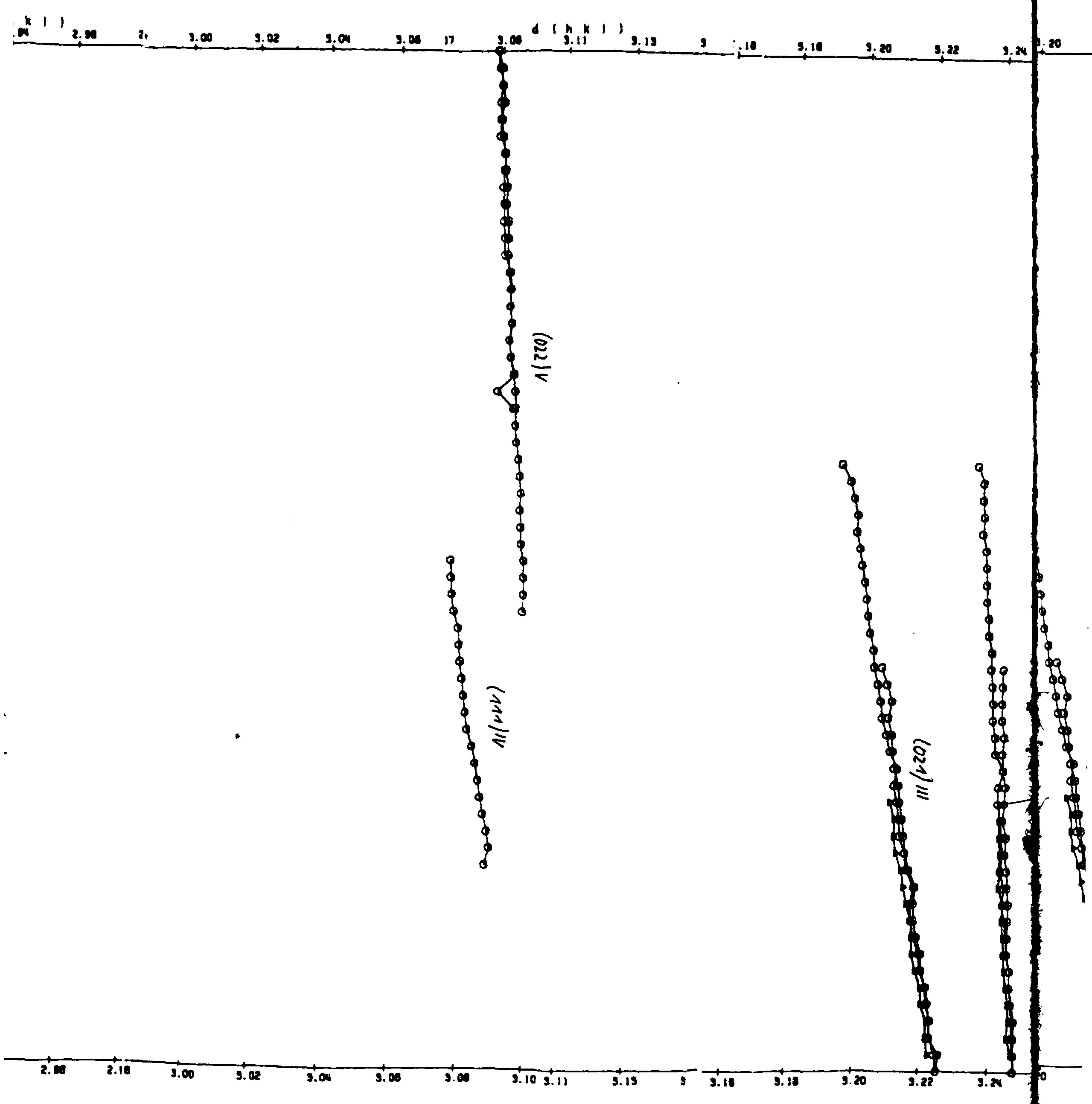


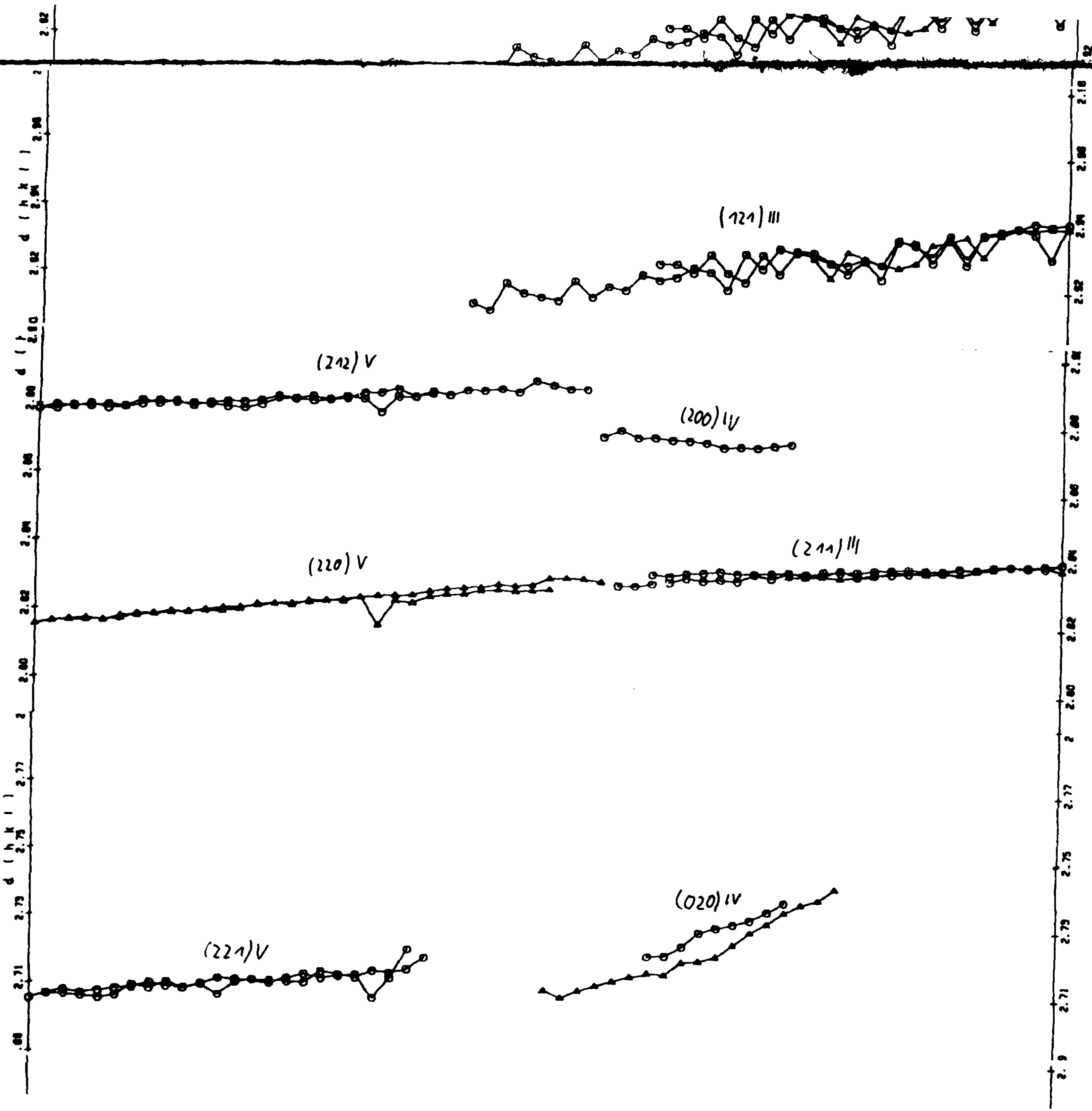


(101) III
(200) V
(110) IV
(111) III
d (h k l)

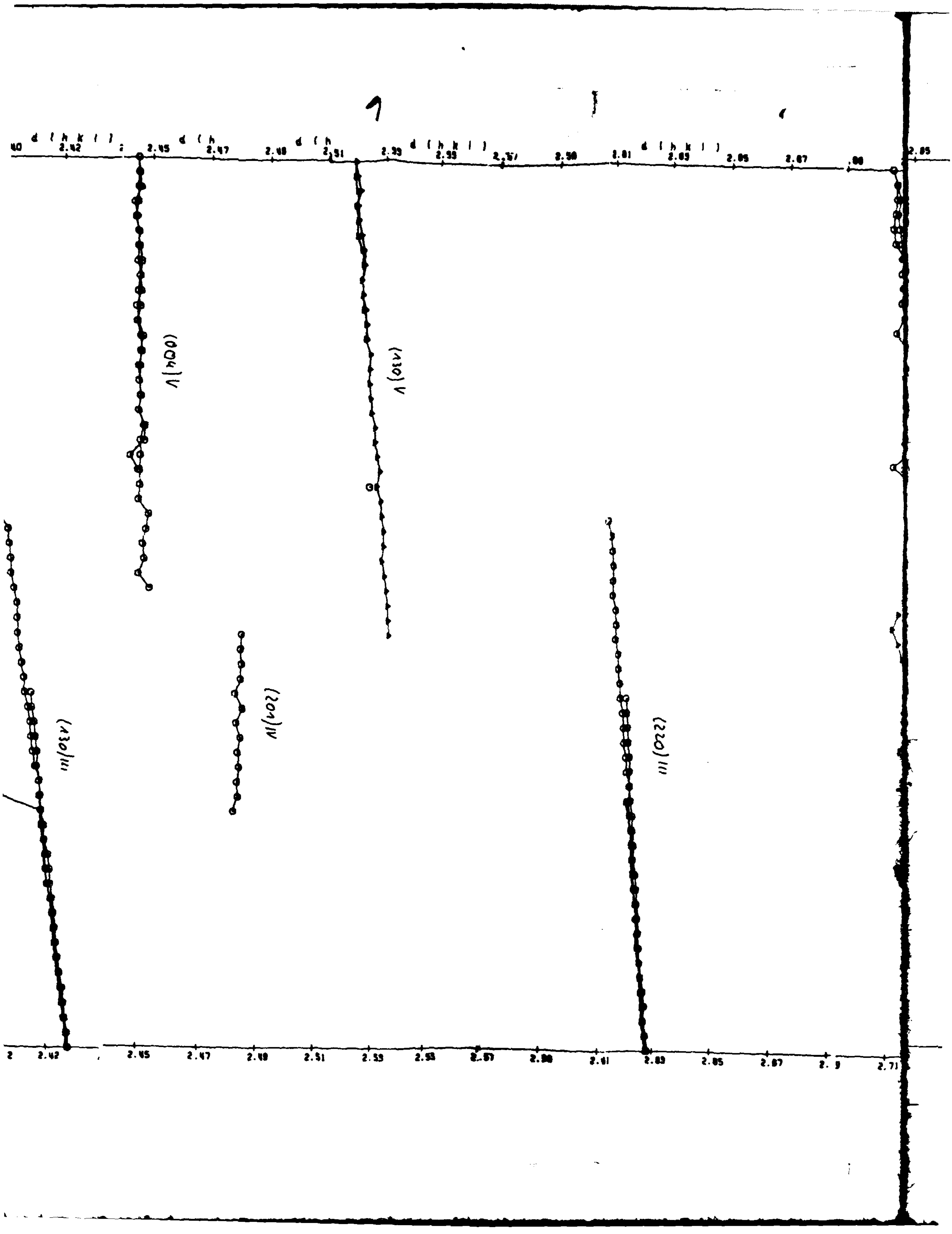
3



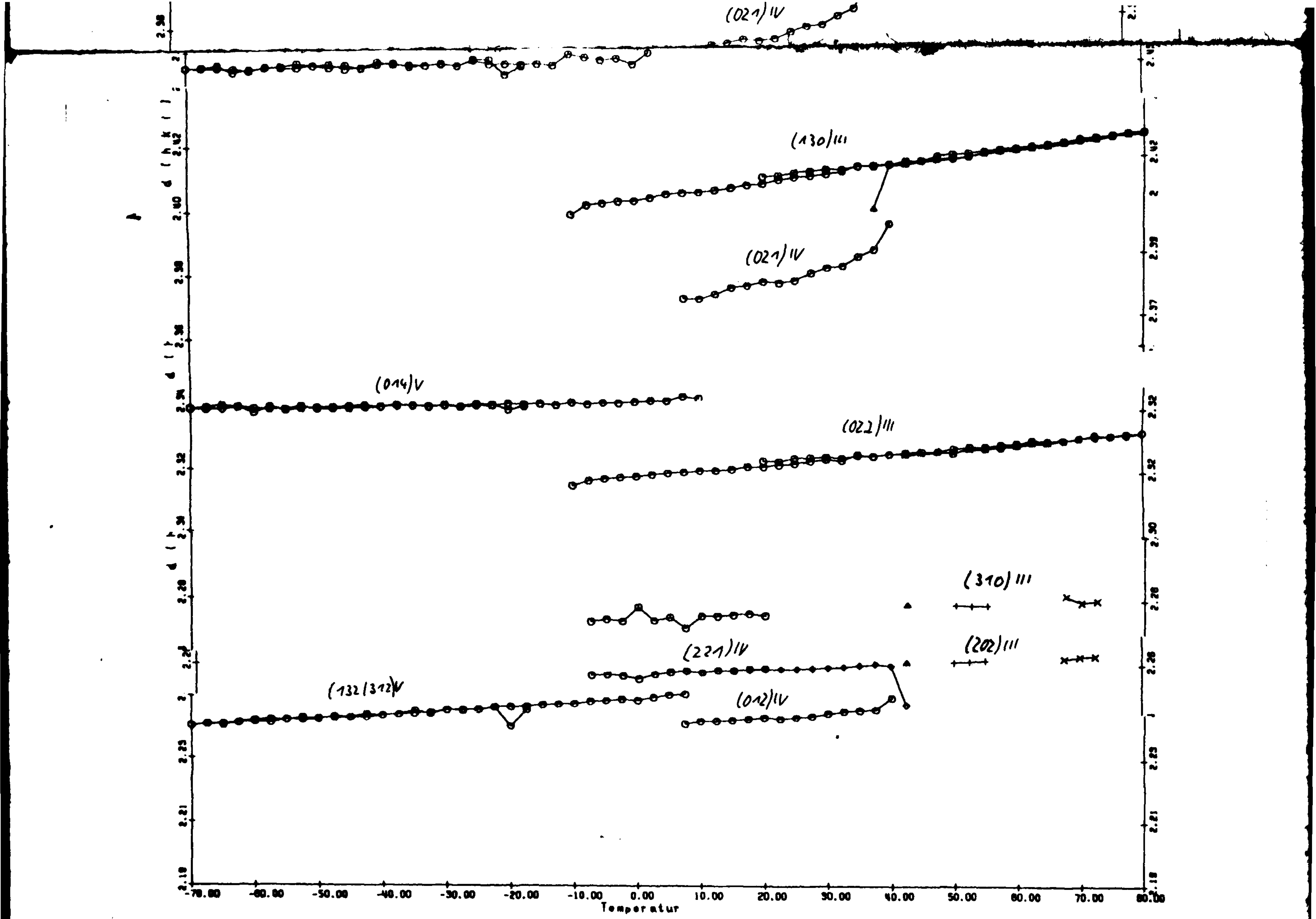

d (h k l)



5



d (h k l)
2.42
2.45
2.47
2.49
2.51
2.53
2.55
2.57
2.59
2.61
2.63
2.65
2.67
2.69
2.71
(004) V
(130) V
(130) III
(201) IV
(220) III

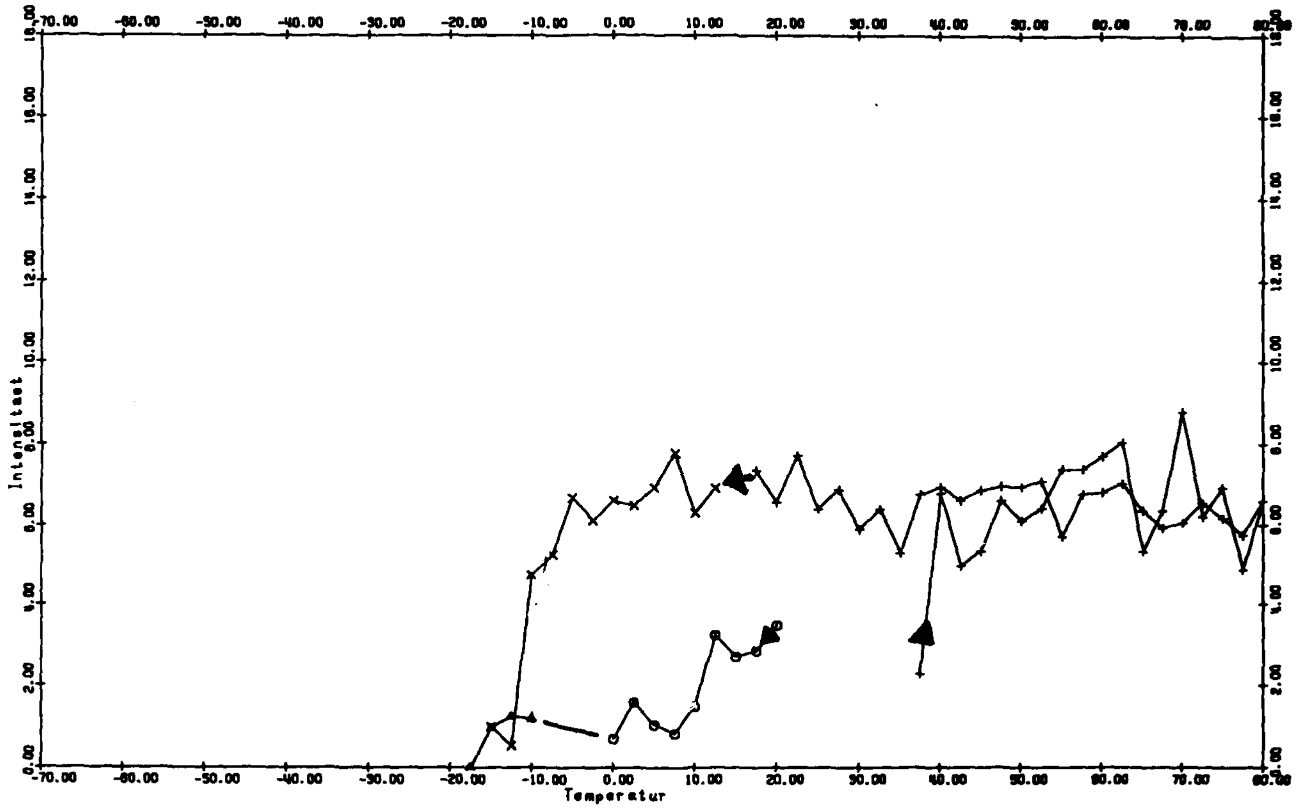


(021)IV
(130)III
(021)IV
(014)V
(022)III
(310)III
(202)III
(221)IV
(012)IV
(132/312)V
Temperatur
d (hkl)

7

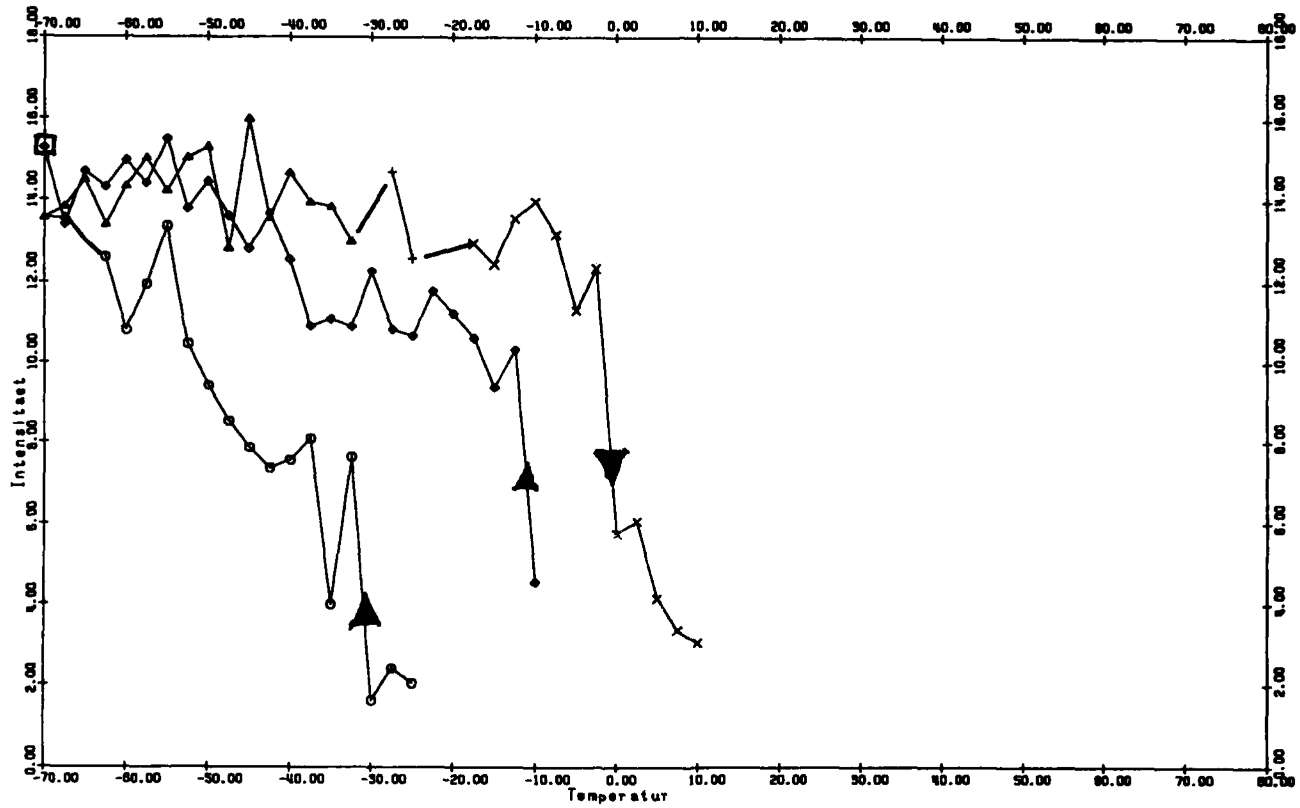
kF 230388

Intensities (220)$_{III}$



kF 230388

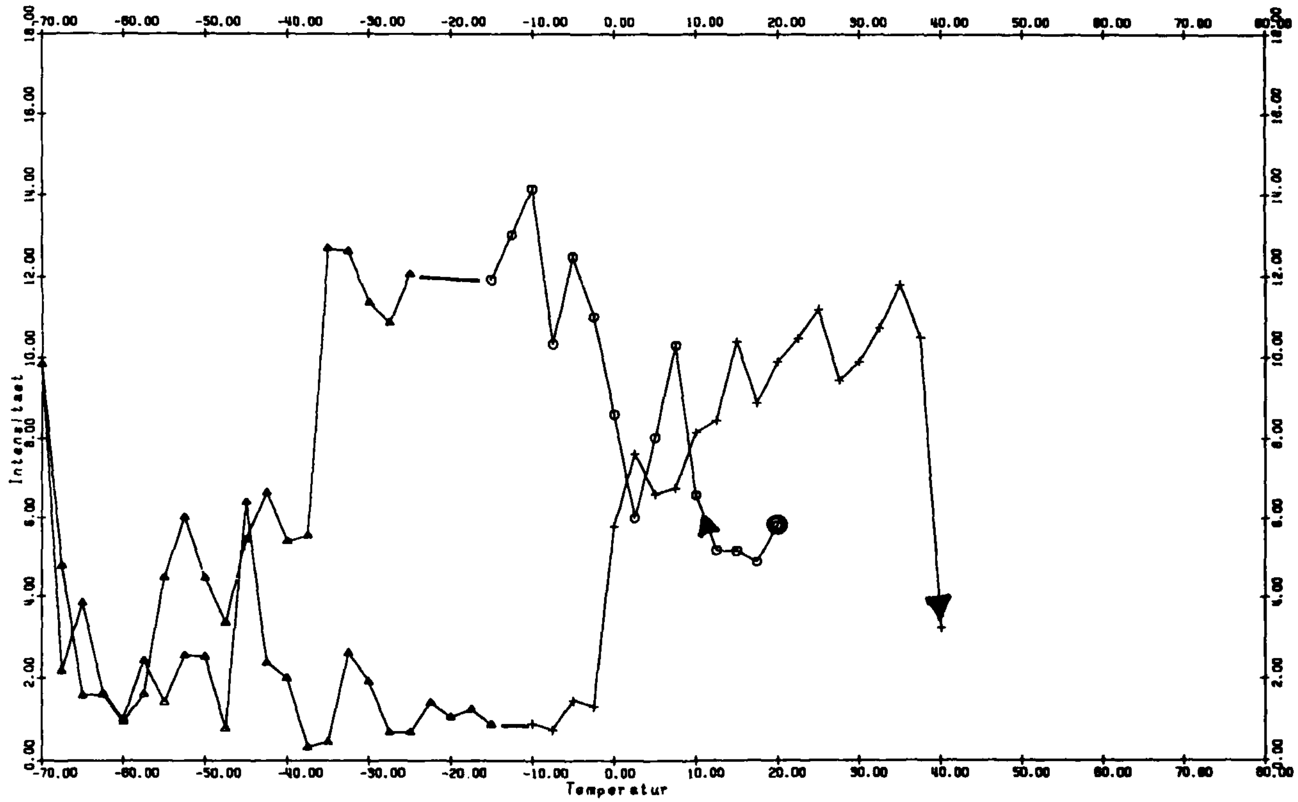
Intensities (022) V (111) ||



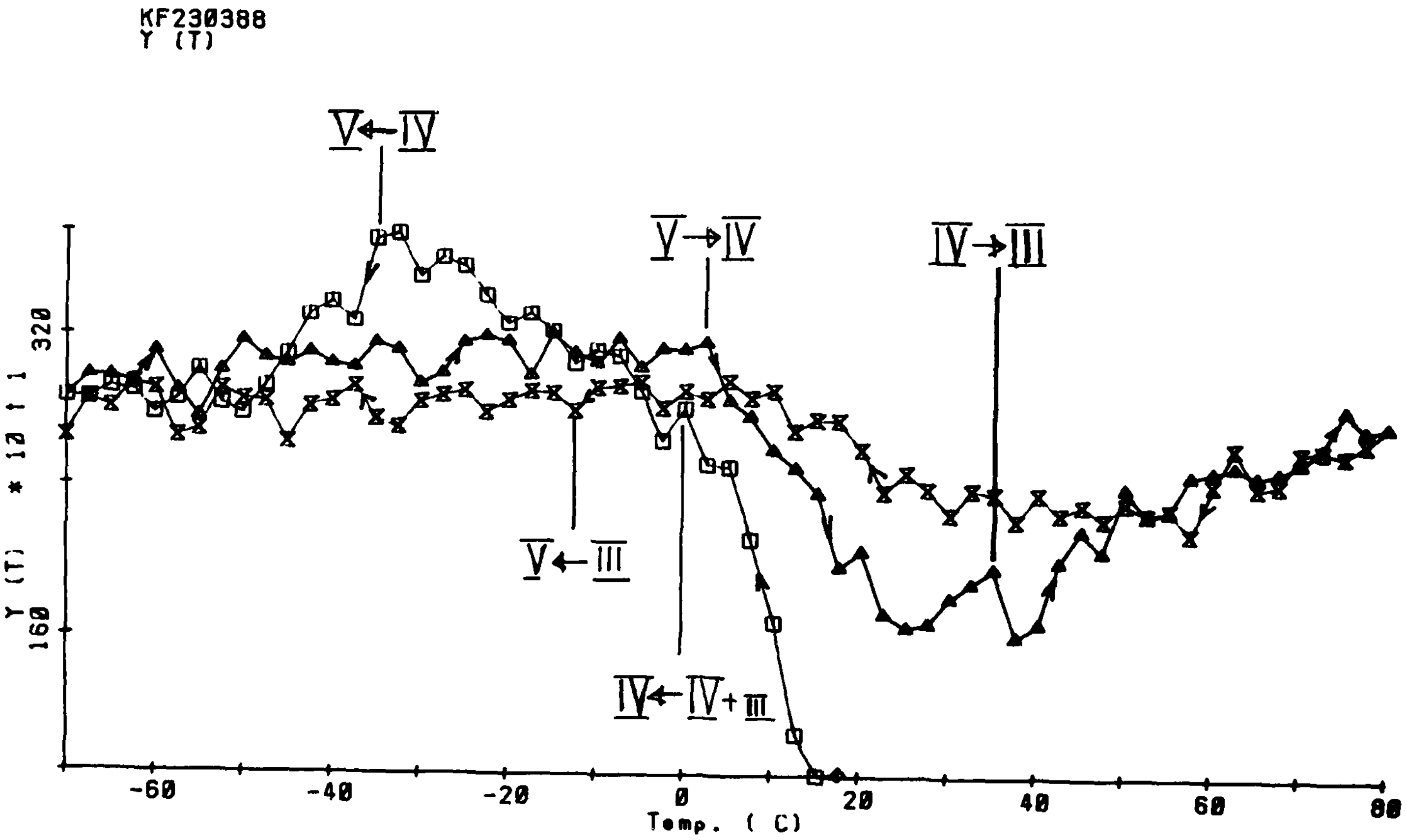
KF 2303 88

(020)$_{IV}$

Intensities



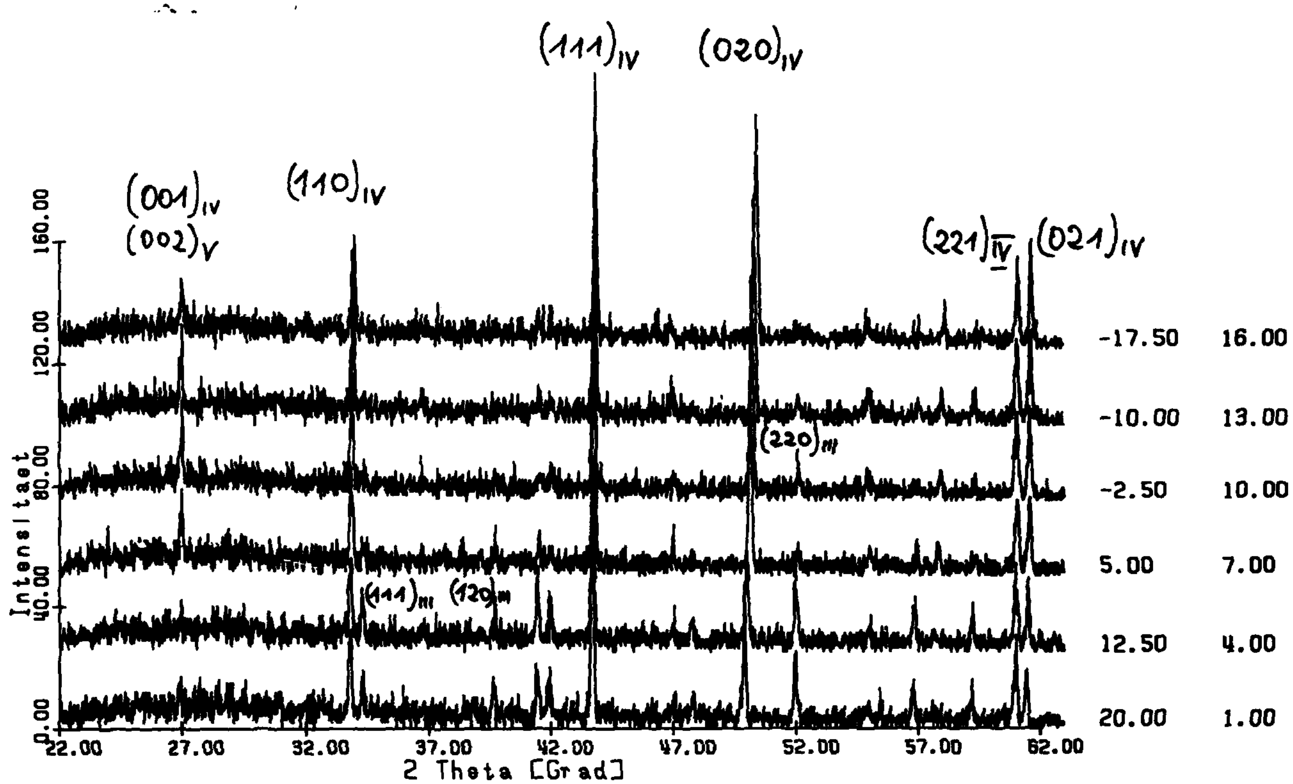
Difference Curve



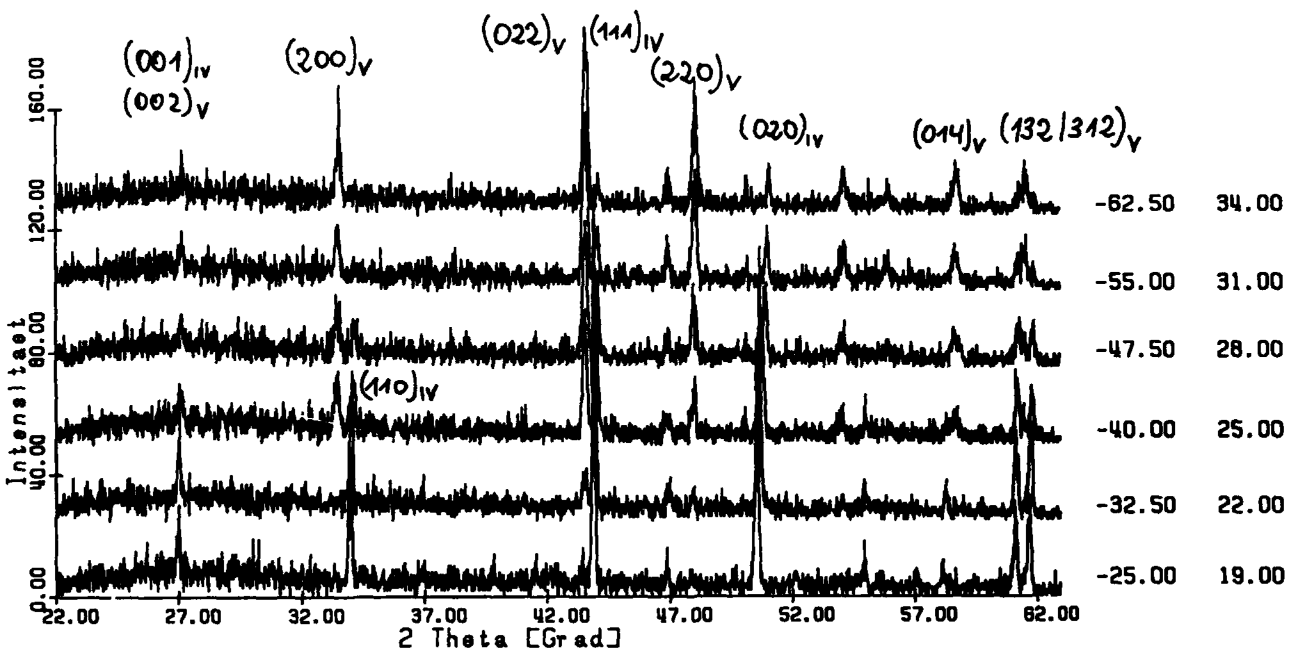
kf230388

20/-70/80/-70 °C

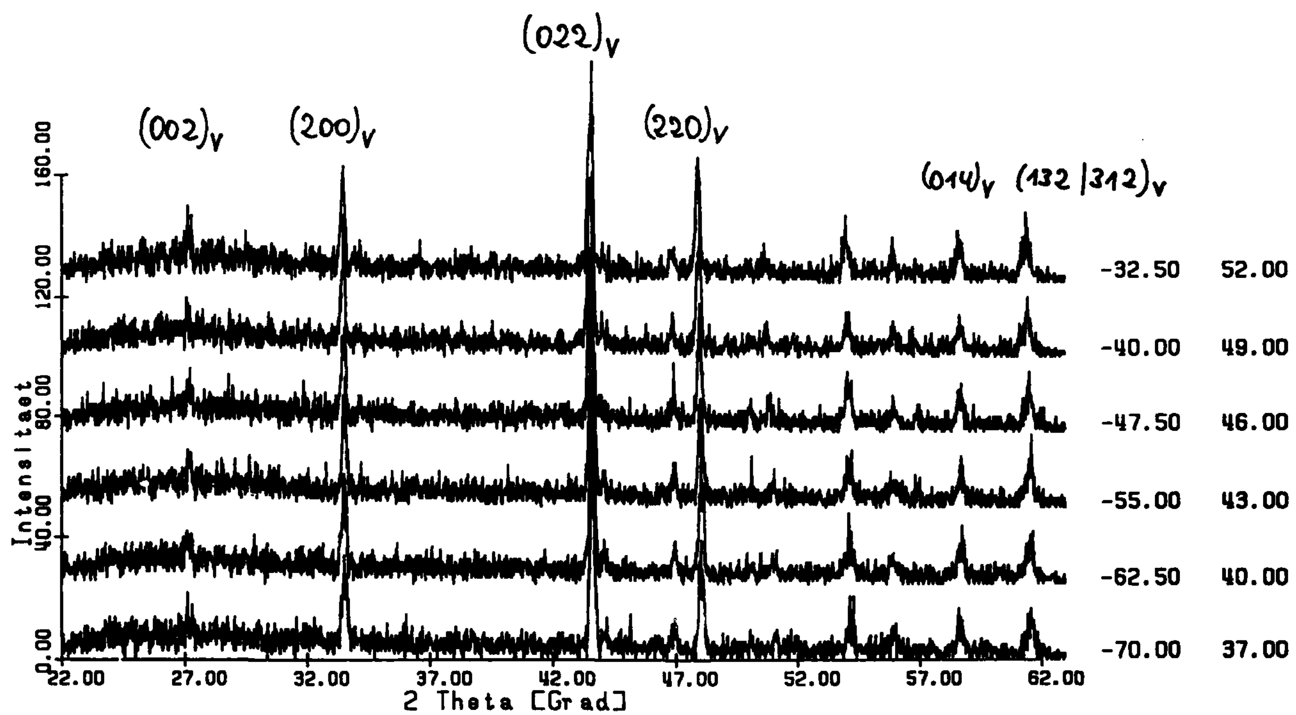
Diffraction Patterns



kf230388

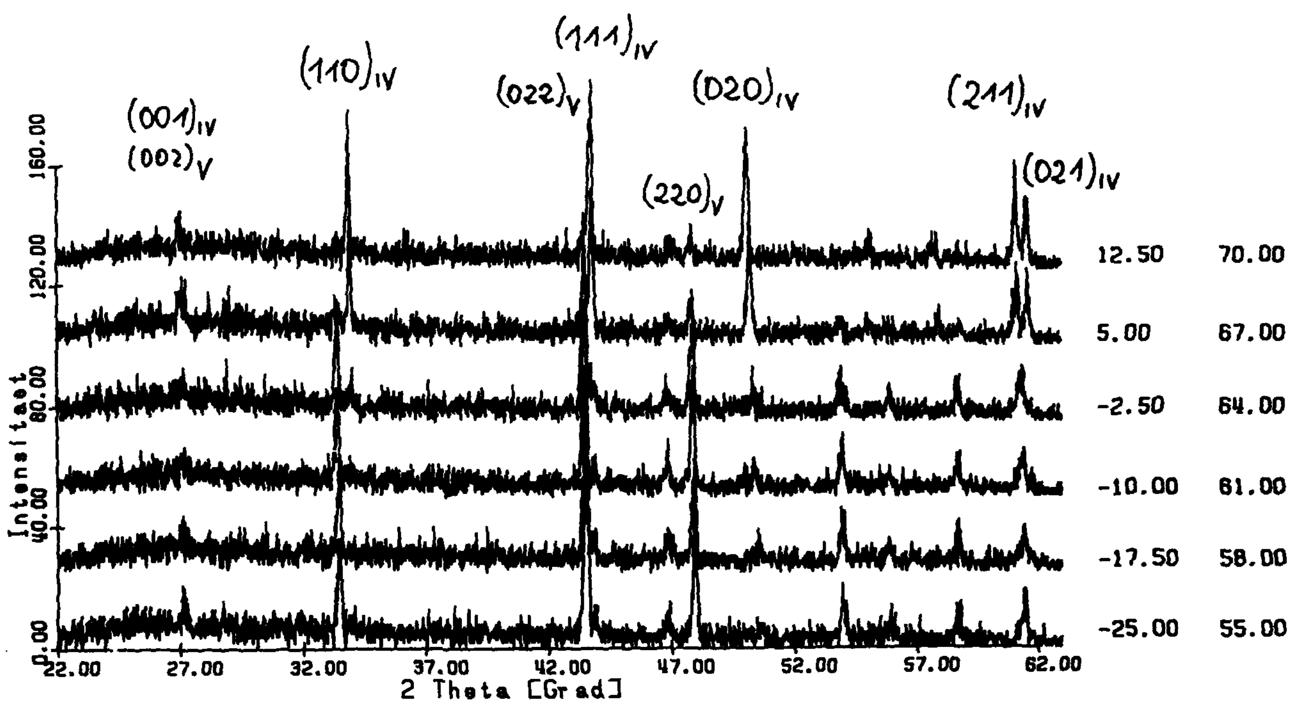

(001)IV
(002)V
(200)V
(022)V
(111)IV
(220)V
(020)IV
(014)V
(132/312)V
(110)IV
Intensitaet
0.00
40.00
80.00
120.00
160.00
22.00
27.00
32.00
37.00
42.00
47.00
52.00
57.00
62.00
2 Theta [Grad]
-62.50 34.00
-55.00 31.00
-47.50 28.00
-40.00 25.00
-32.50 22.00
-25.00 19.00

kf230388



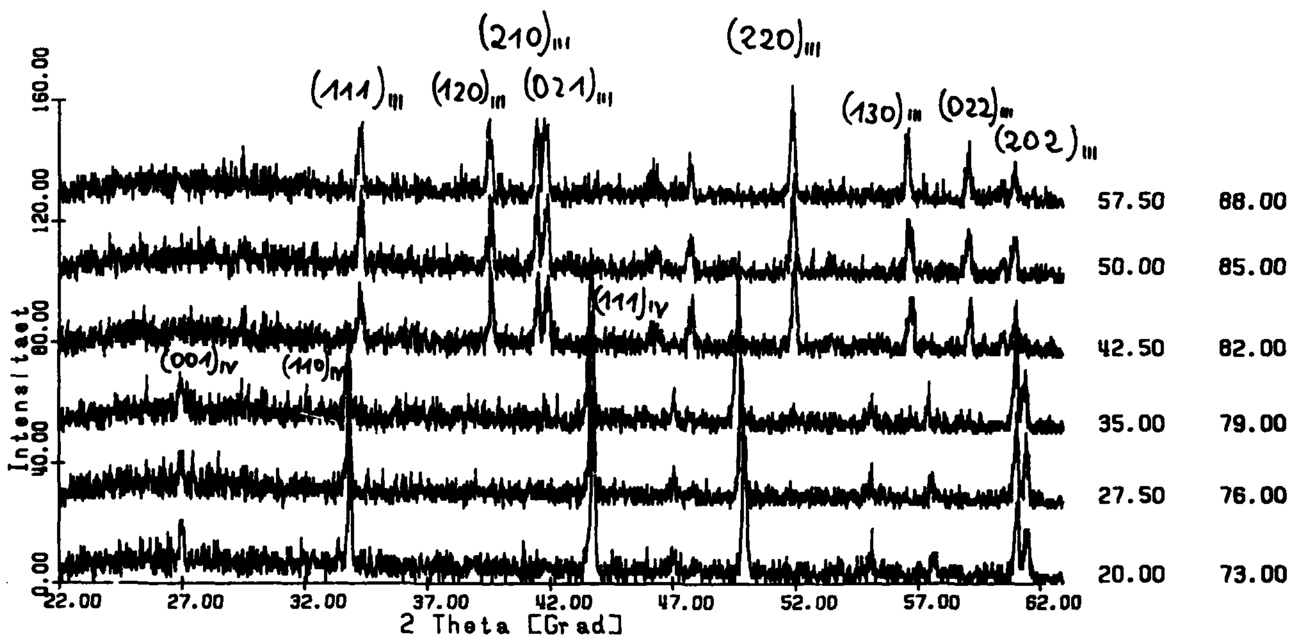
$(002)_V$
$(200)_V$
$(022)_V$
$(220)_V$
$(014)_V$
$(132|312)_V$
160.00
120.00
80.00
40.00
0.00
Intensitaet
22.00
27.00
32.00
37.00
42.00
47.00
52.00
57.00
62.00
2 Theta [Grad]
-32.50 52.00
-40.00 49.00
-47.50 46.00
-55.00 43.00
-62.50 40.00
-70.00 37.00

kf230388



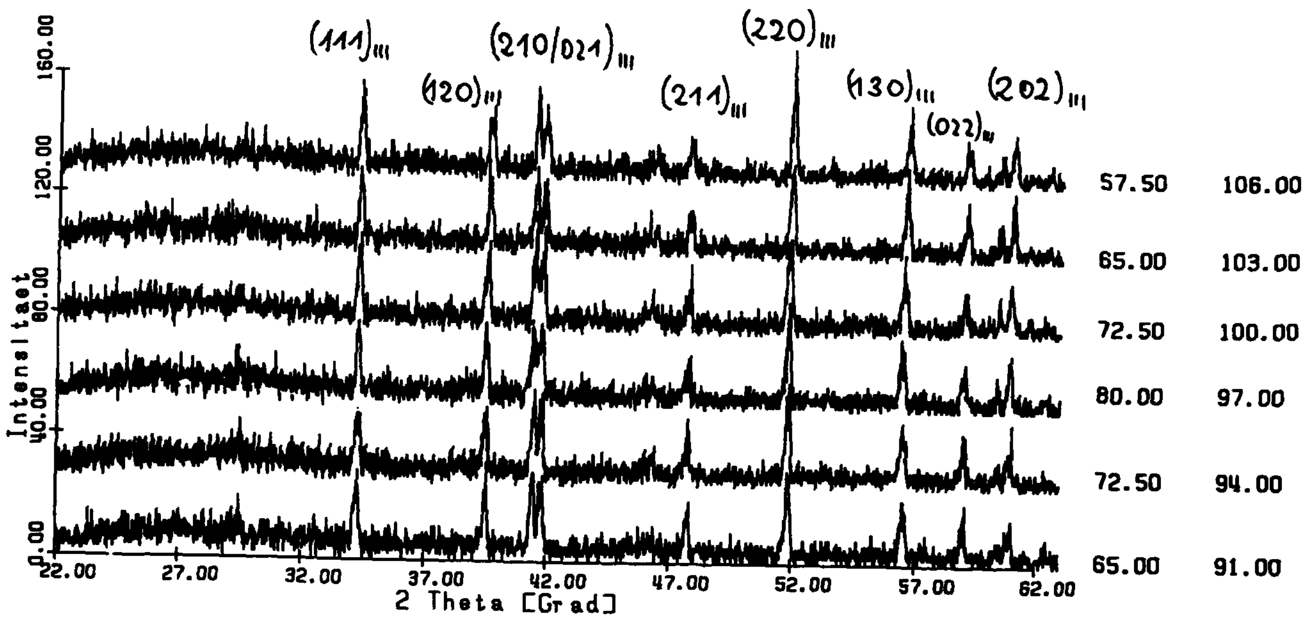
(001)IV
(002)V
(110)IV
(022)V
(111)IV
(020)IV
(220)V
(211)IV
(021)IV
160.00
120.00
80.00
40.00
0.00
Intensitaet
22.00
27.00
32.00
37.00
42.00
47.00
52.00
57.00
62.00
2 Theta [Grad]
12.50 70.00
5.00 67.00
-2.50 64.00
-10.00 61.00
-17.50 58.00
-25.00 55.00

kf230388



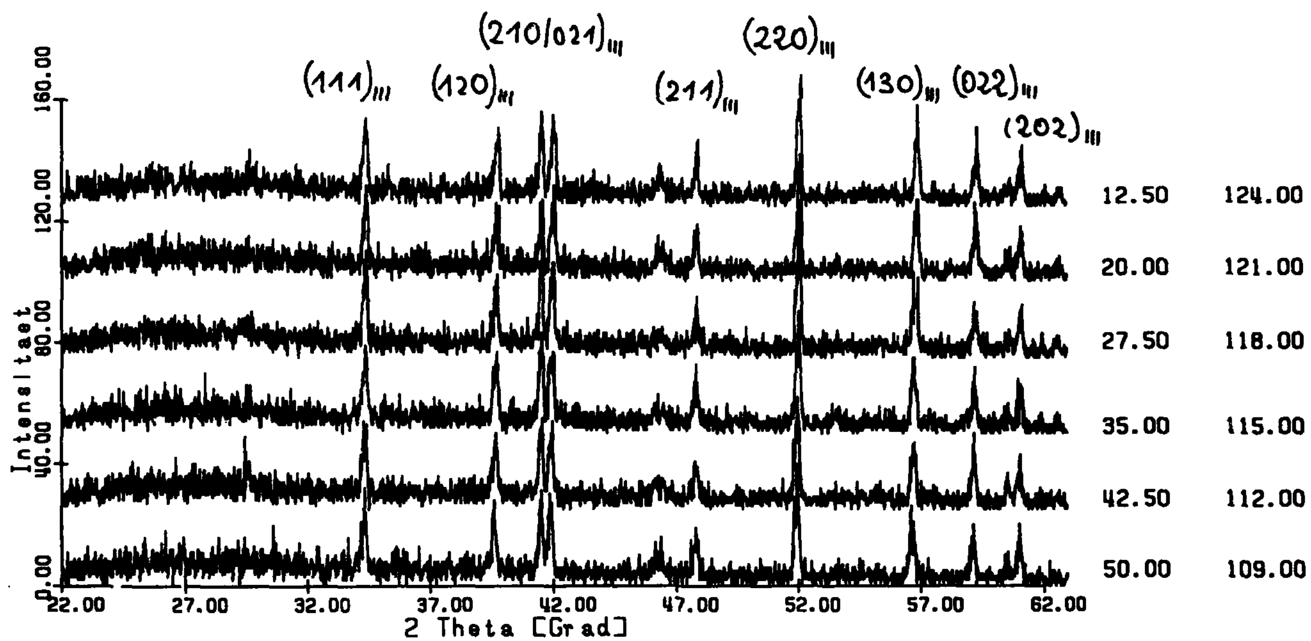
(111)III
(120)III
(210)III
(021)III
(220)III
(130)III
(022)III
(202)III
(001)IV
(110)IV
(111)IV
Intensitaet
0.00
40.00
80.00
120.00
160.00
22.00
27.00
32.00
37.00
42.00
47.00
52.00
57.00
62.00
2 Theta [Grad]
57.50 88.00
50.00 85.00
42.50 82.00
35.00 79.00
27.50 76.00
20.00 73.00

kf230388

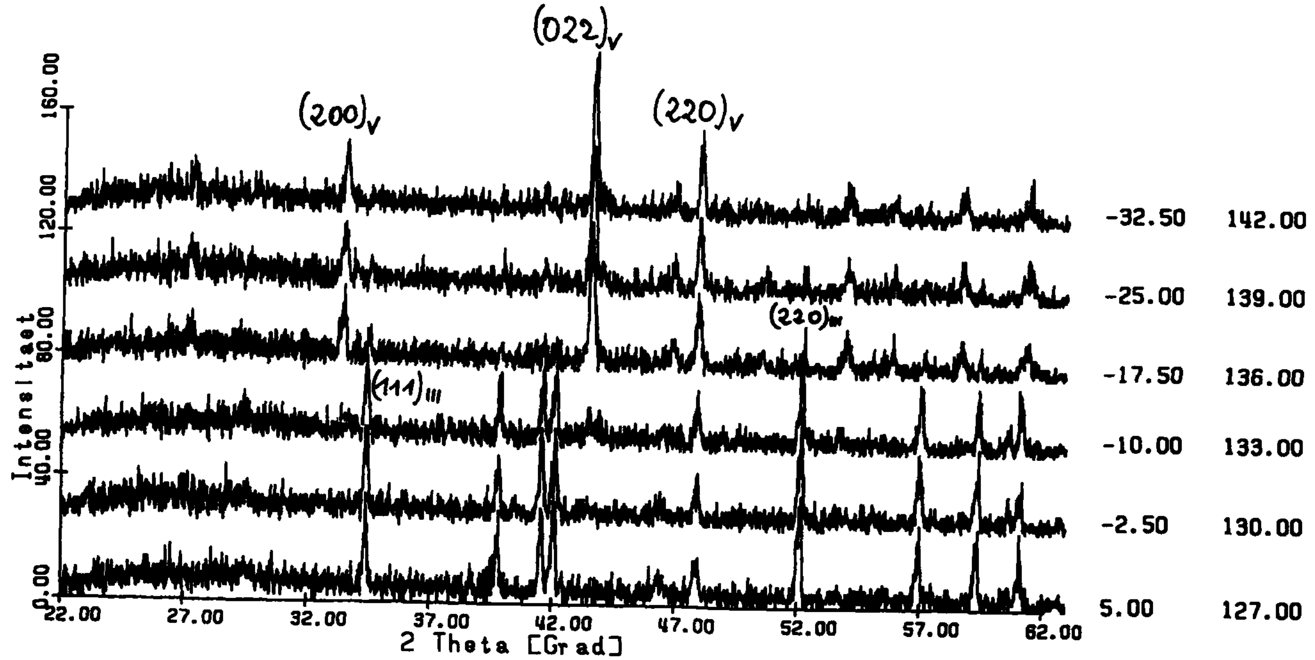


(111)III
(120)III
(210/021)III
(211)III
(220)III
(130)III
(022)III
(202)III
160.00
120.00
80.00
40.00
0.00
Intensitaet
22.00
27.00
32.00
37.00
42.00
47.00
52.00
57.00
62.00
2 Theta [Grad]
57.50 106.00
65.00 103.00
72.50 100.00
80.00 97.00
72.50 94.00
65.00 91.00

kf230388

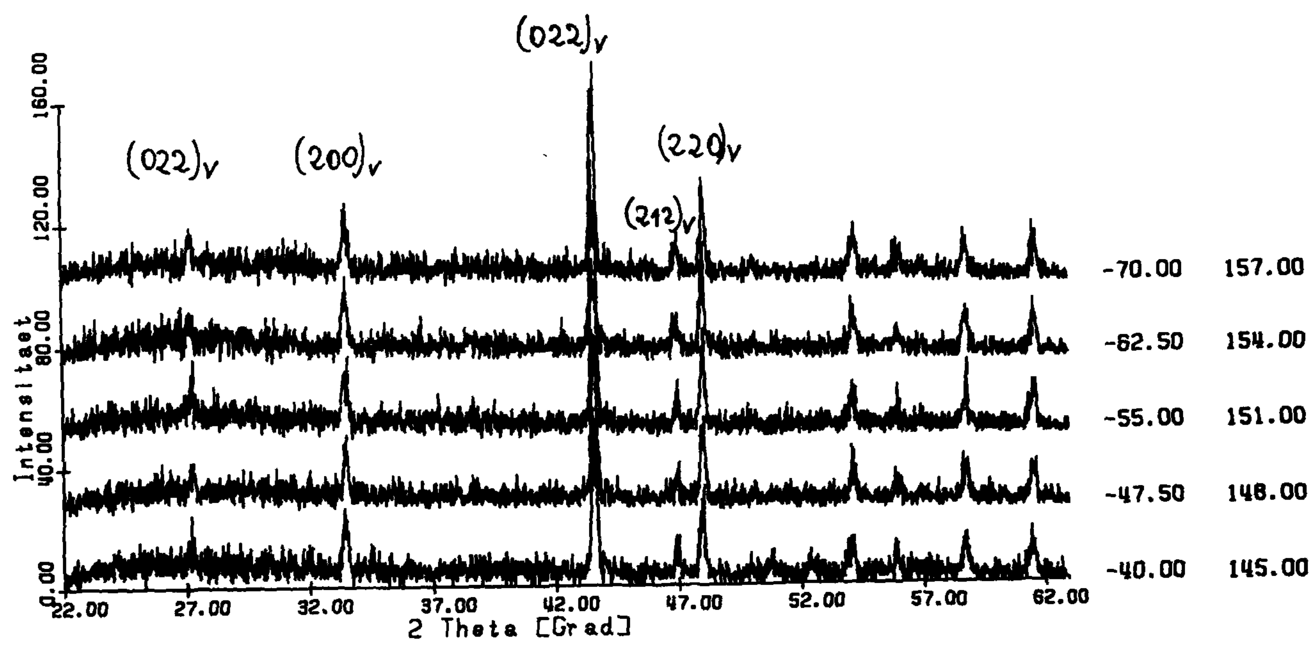

(111)III
(120)III
(210/021)III
(211)III
(220)III
(130)III
(022)III
(202)III
Intensitaet
0.00
40.00
80.00
120.00
160.00
22.00
27.00
32.00
37.00
42.00
47.00
52.00
57.00
62.00
2 Theta [Grad]
12.50
124.00
20.00
121.00
27.50
118.00
35.00
115.00
42.50
112.00
50.00
109.00

kf230388



(022)V
(200)V
(220)V
(220)III
(111)III
160.00
120.00
80.00
40.00
0.00
Intensitaet
22.00
27.00
32.00
37.00
42.00
47.00
52.00
57.00
62.00
2 Theta [Grad]
-32.50
142.00
-25.00
139.00
-17.50
136.00
-10.00
133.00
-2.50
130.00
5.00
127.00

kf230388



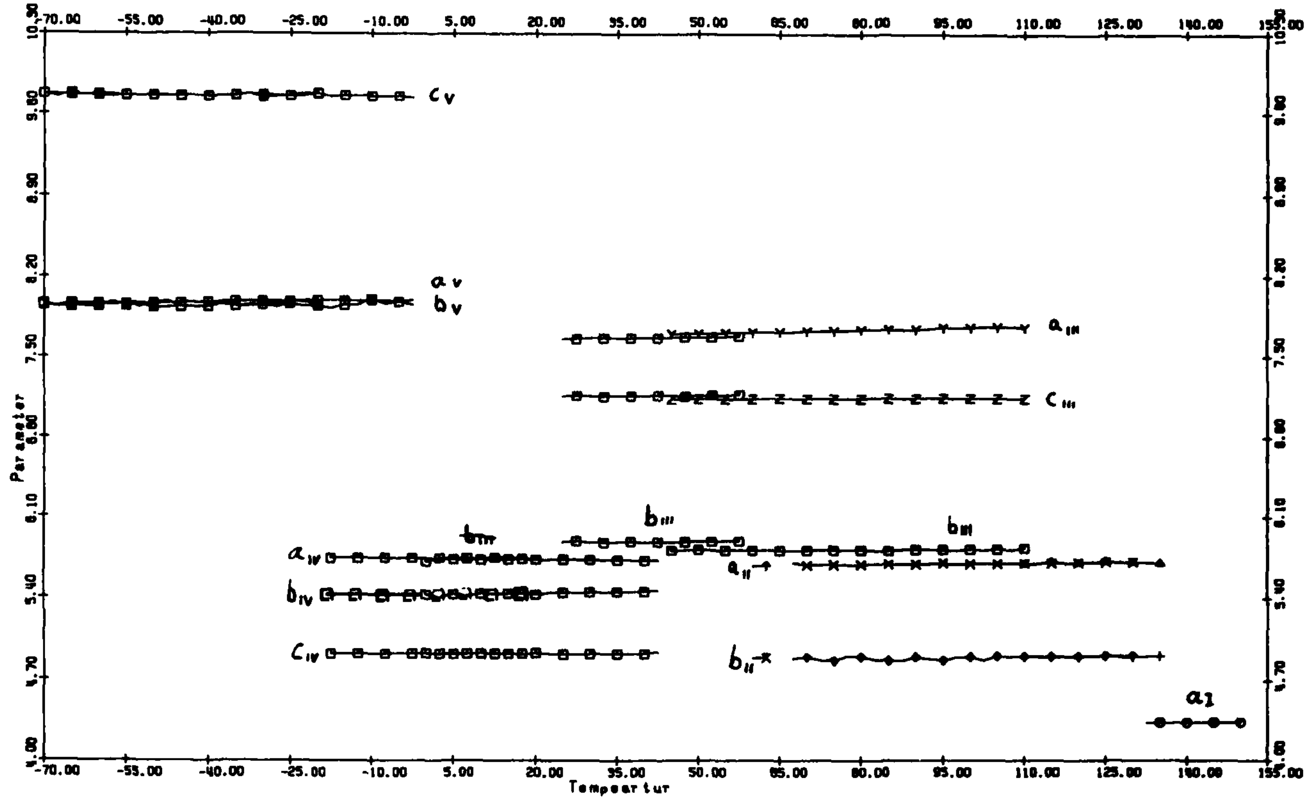
(022)v
(022)v
(200)v
(212)v
(220)v
160.00
120.00
80.00
40.00
0.00
Intensitaet
22.00
27.00
32.00
37.00
42.00
47.00
52.00
57.00
62.00
2 Theta [Grad]
-70.00 157.00
-62.50 154.00
-55.00 151.00
-47.50 148.00
-40.00 145.00

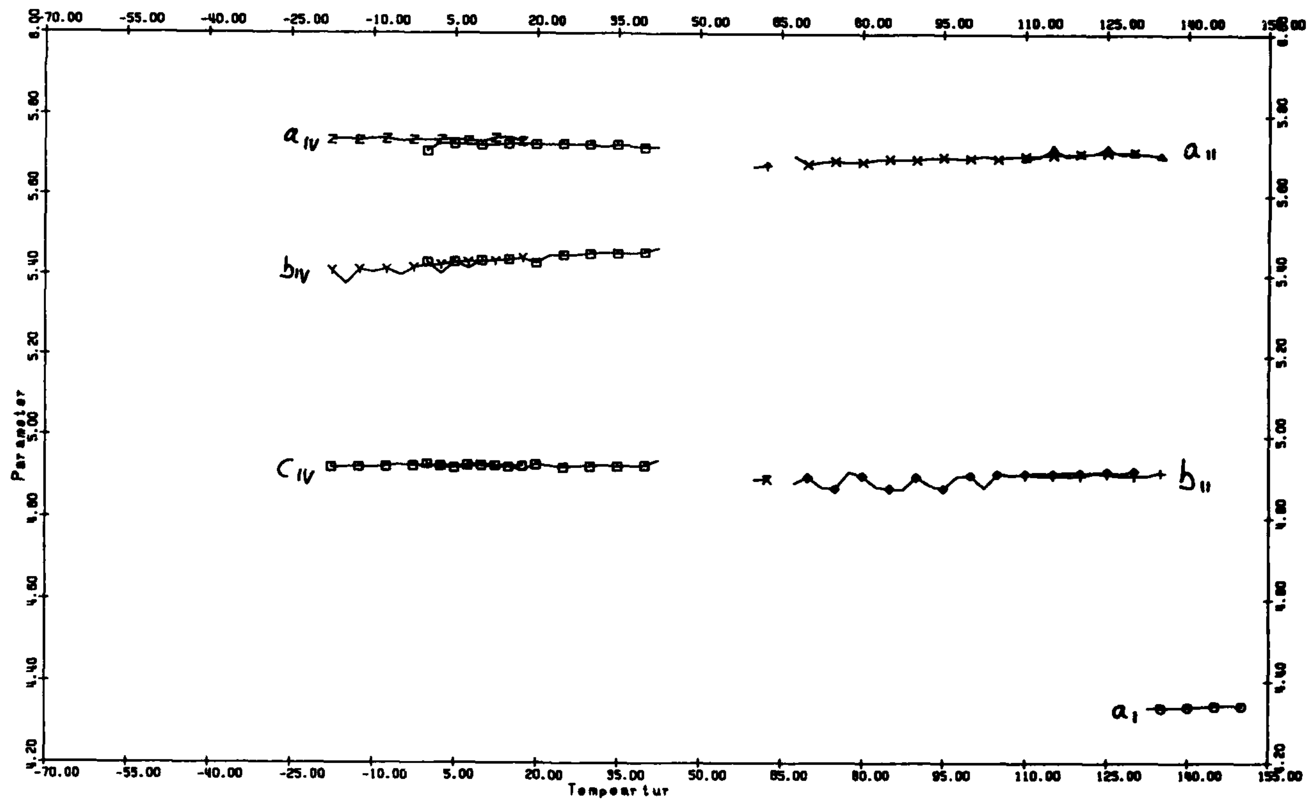
Series
KF 290388

Temperature
Program
20/—70/150/20

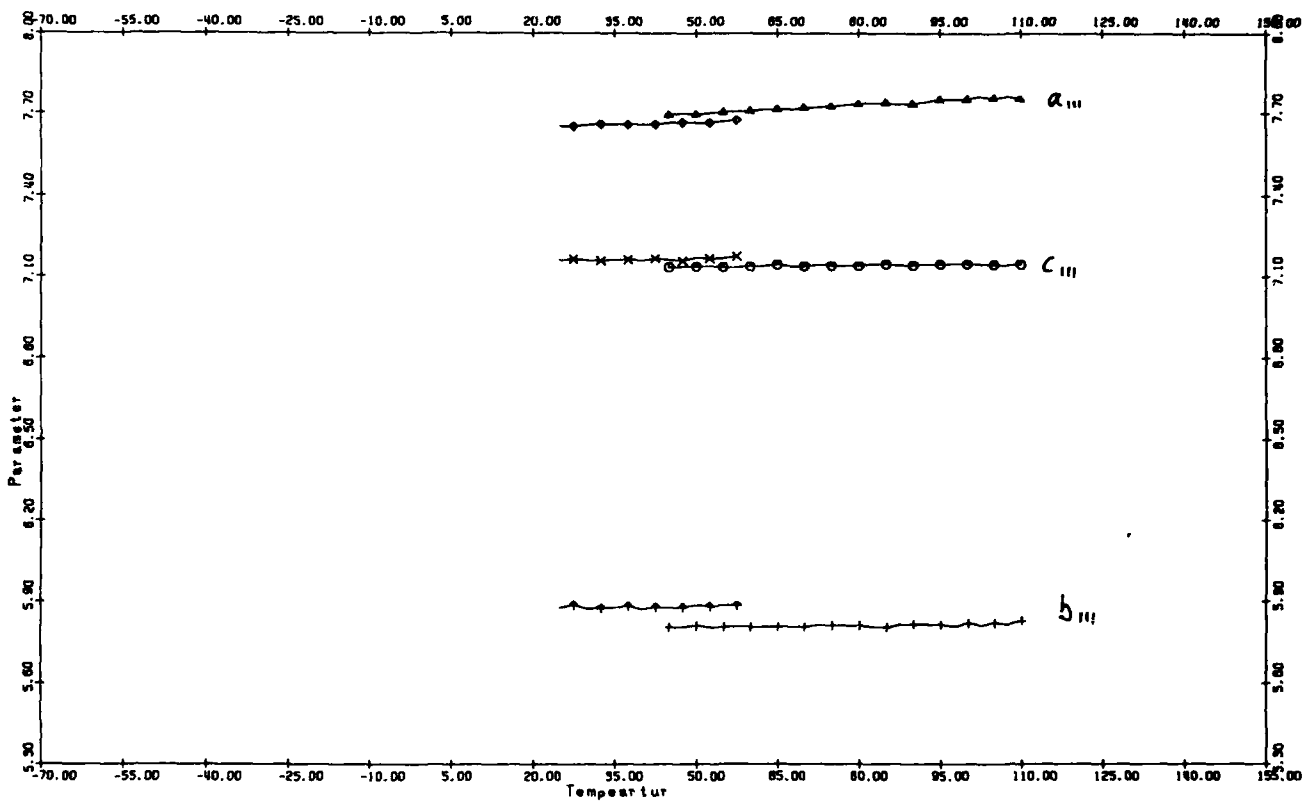
KF 290388
20/-70/150/20

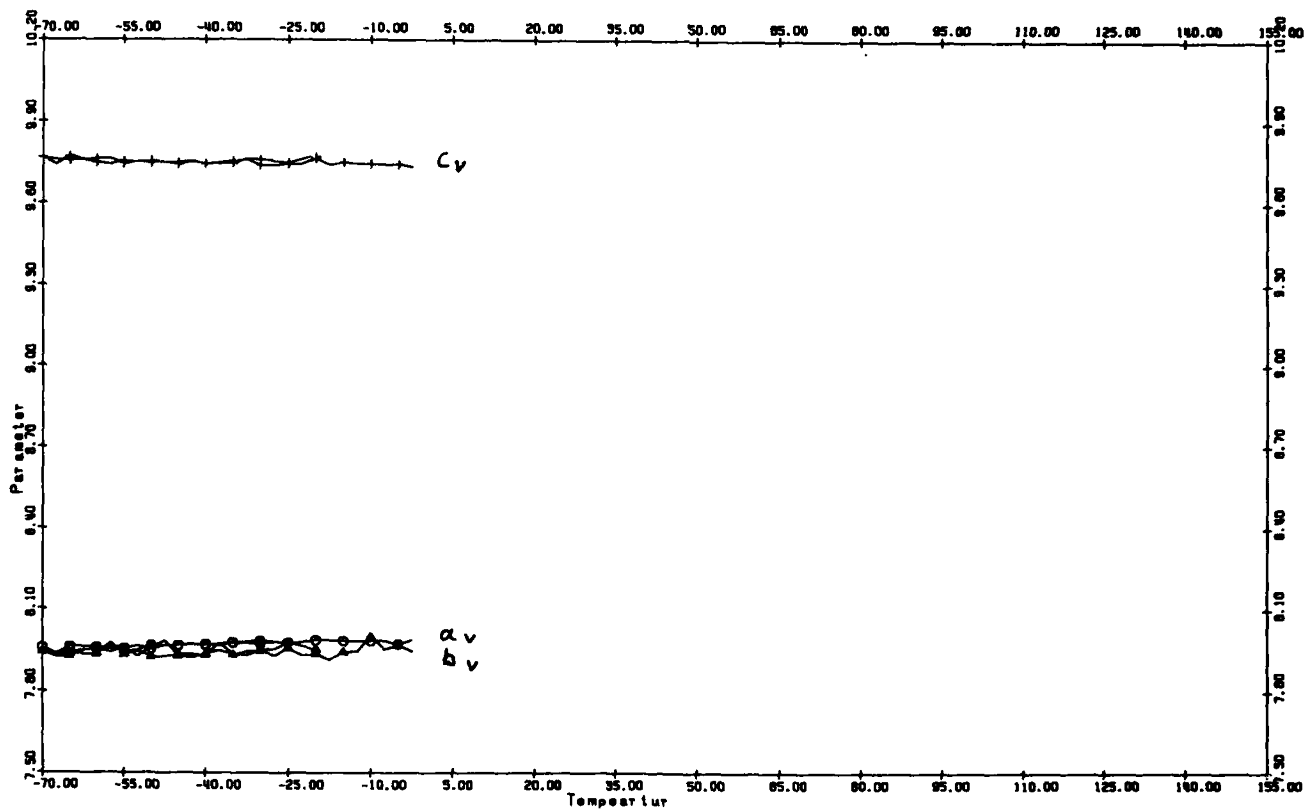
Lattice Parameters





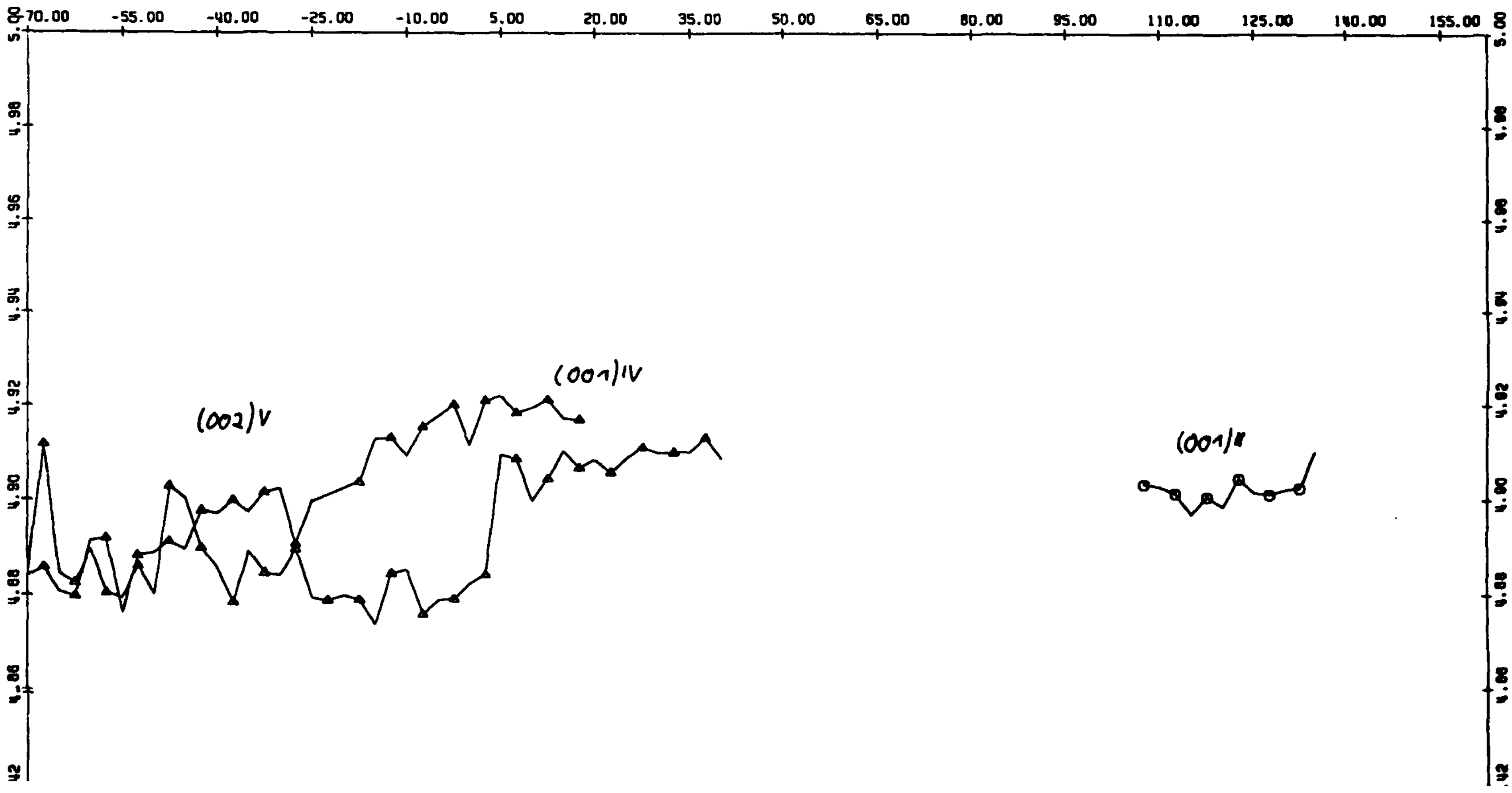
Parameter
Temperatur
-70.00
-55.00
-40.00
-25.00
-10.00
5.00
20.00
35.00
50.00
65.00
80.00
95.00
110.00
125.00
140.00
155.00
4.20
4.40
4.60
4.80
5.00
5.20
5.40
5.60
5.80
6.00
a_{IV}
b_{IV}
c_{IV}
a_{II}
b_{II}
a_{I}

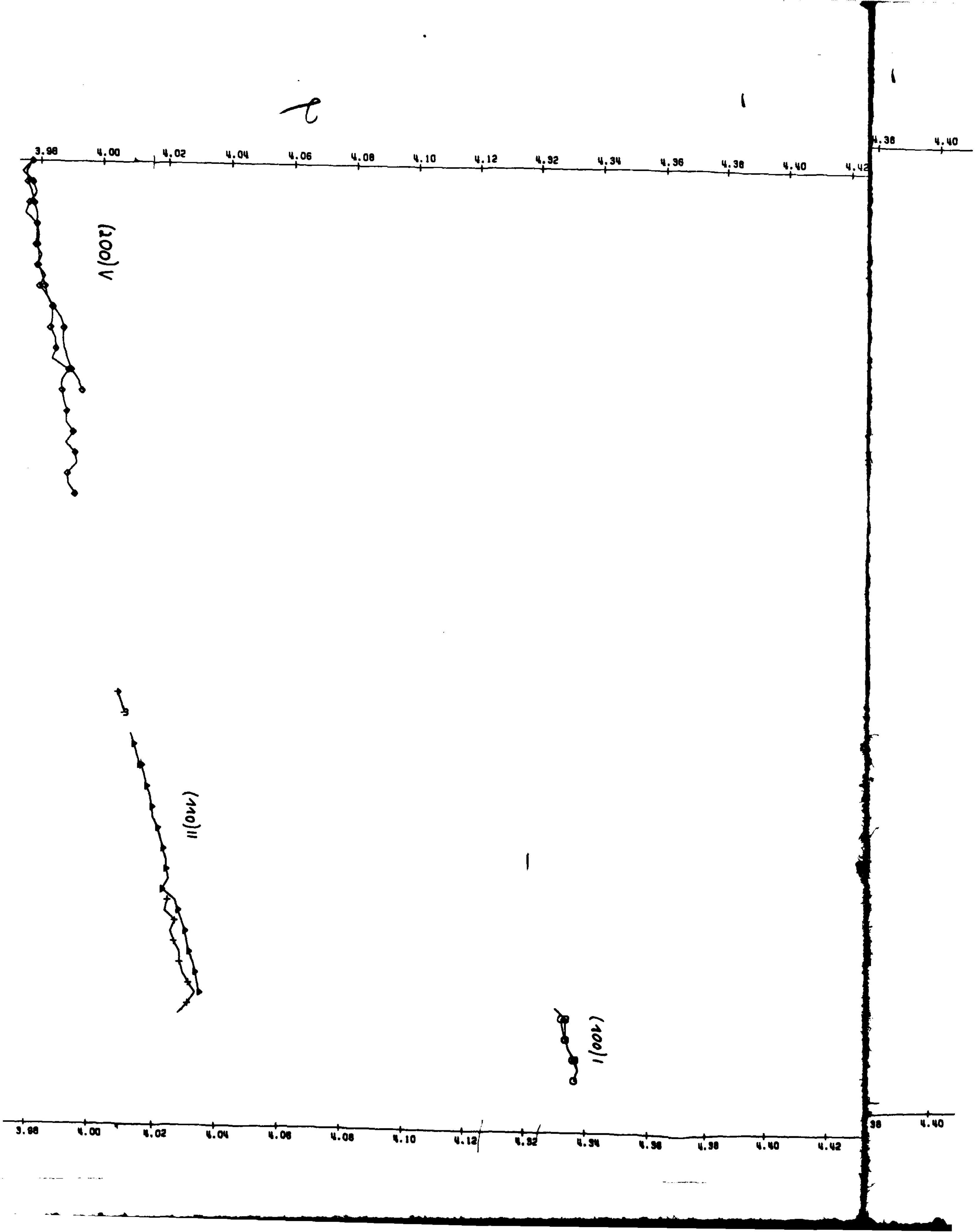

-70.00
-55.00
-40.00
-25.00
-10.00
5.00
20.00
35.00
50.00
65.00
80.00
95.00
110.00
125.00
140.00
155.00
Tempeartur
8.00
7.70
7.40
7.10
6.80
6.50
6.20
5.90
5.60
5.30
Parameter
a111
c111
b111


Parameter
Temperatur
av
bv
cv

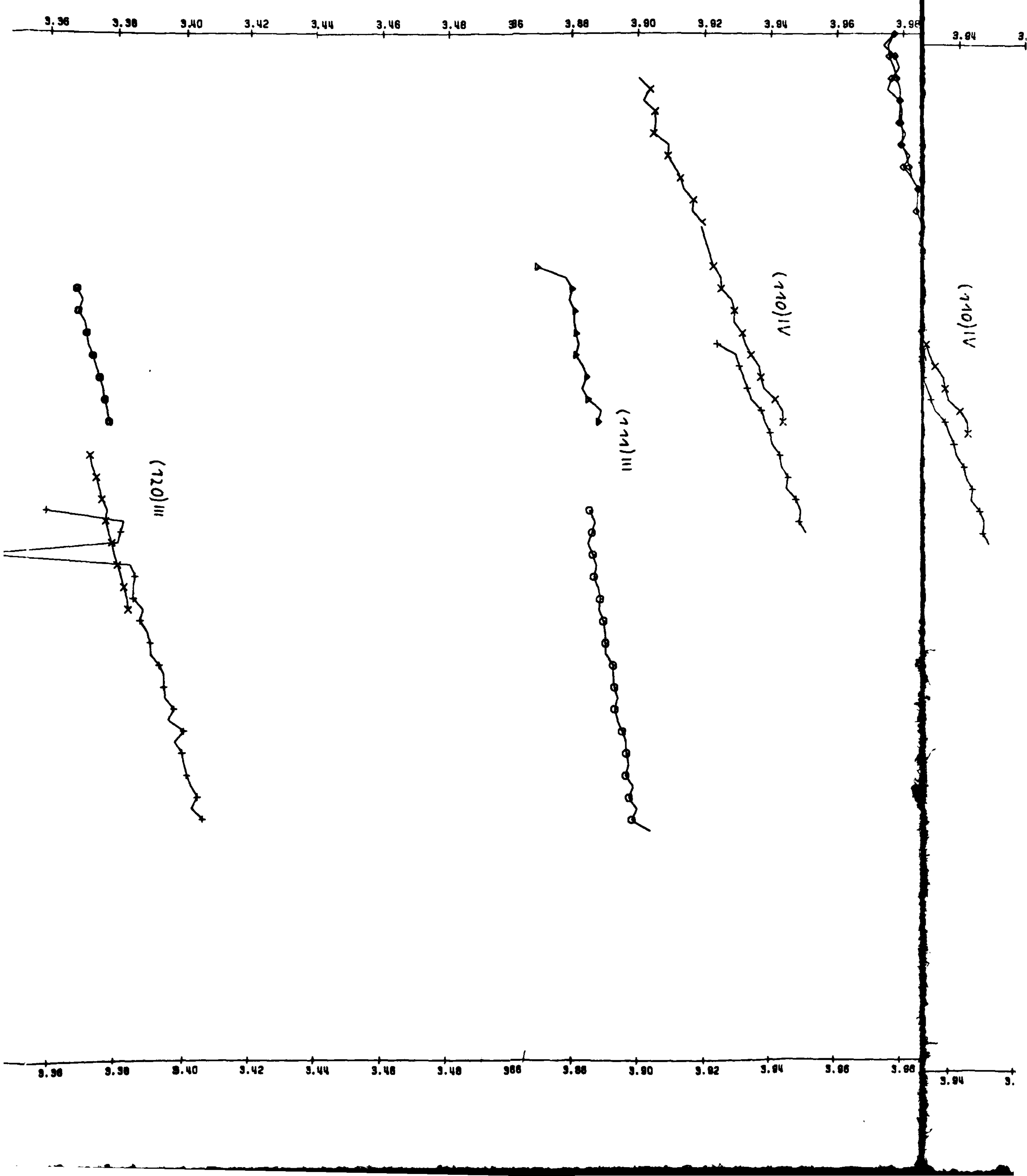
K7 290388

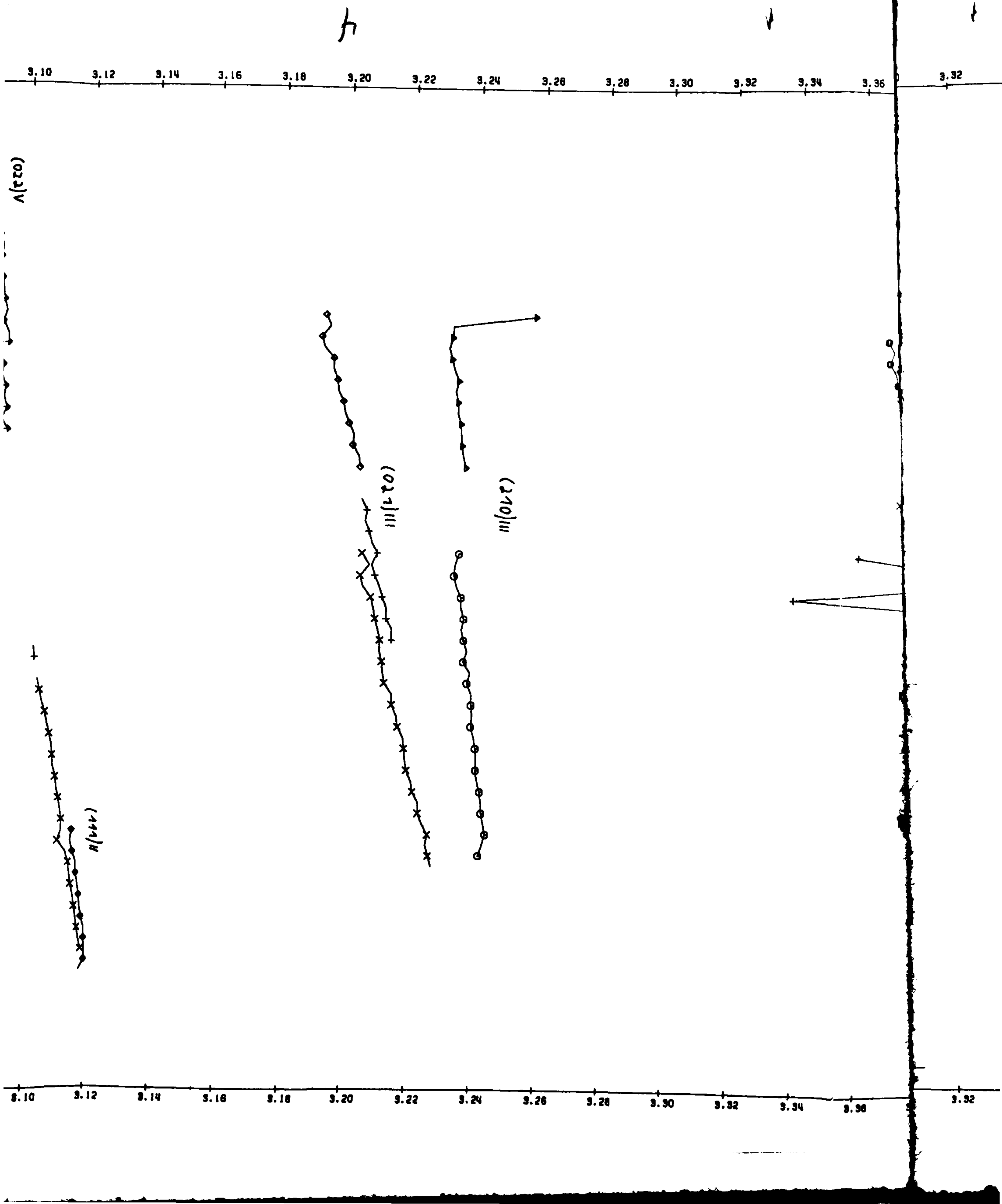
Lattice Distances (d)

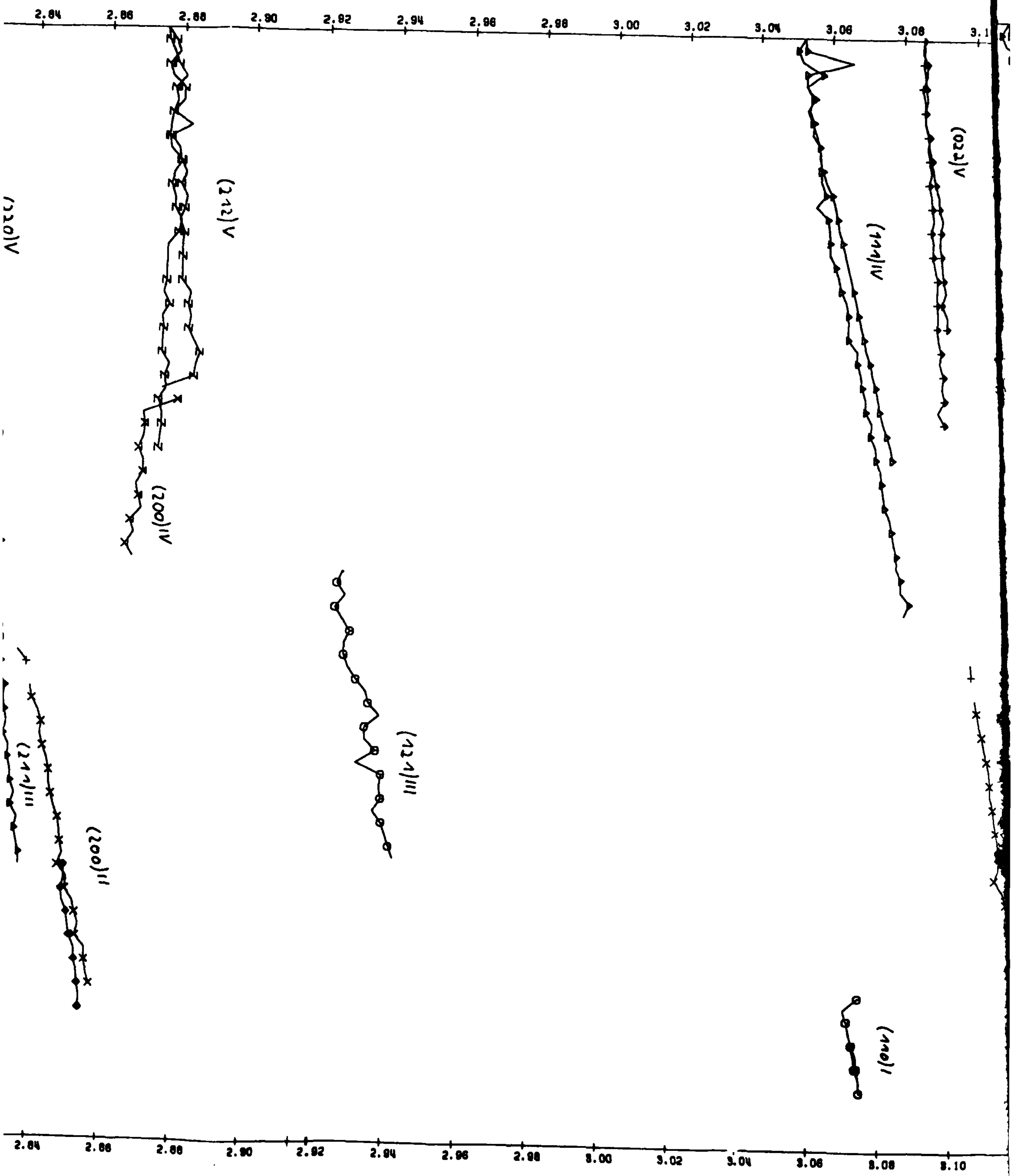


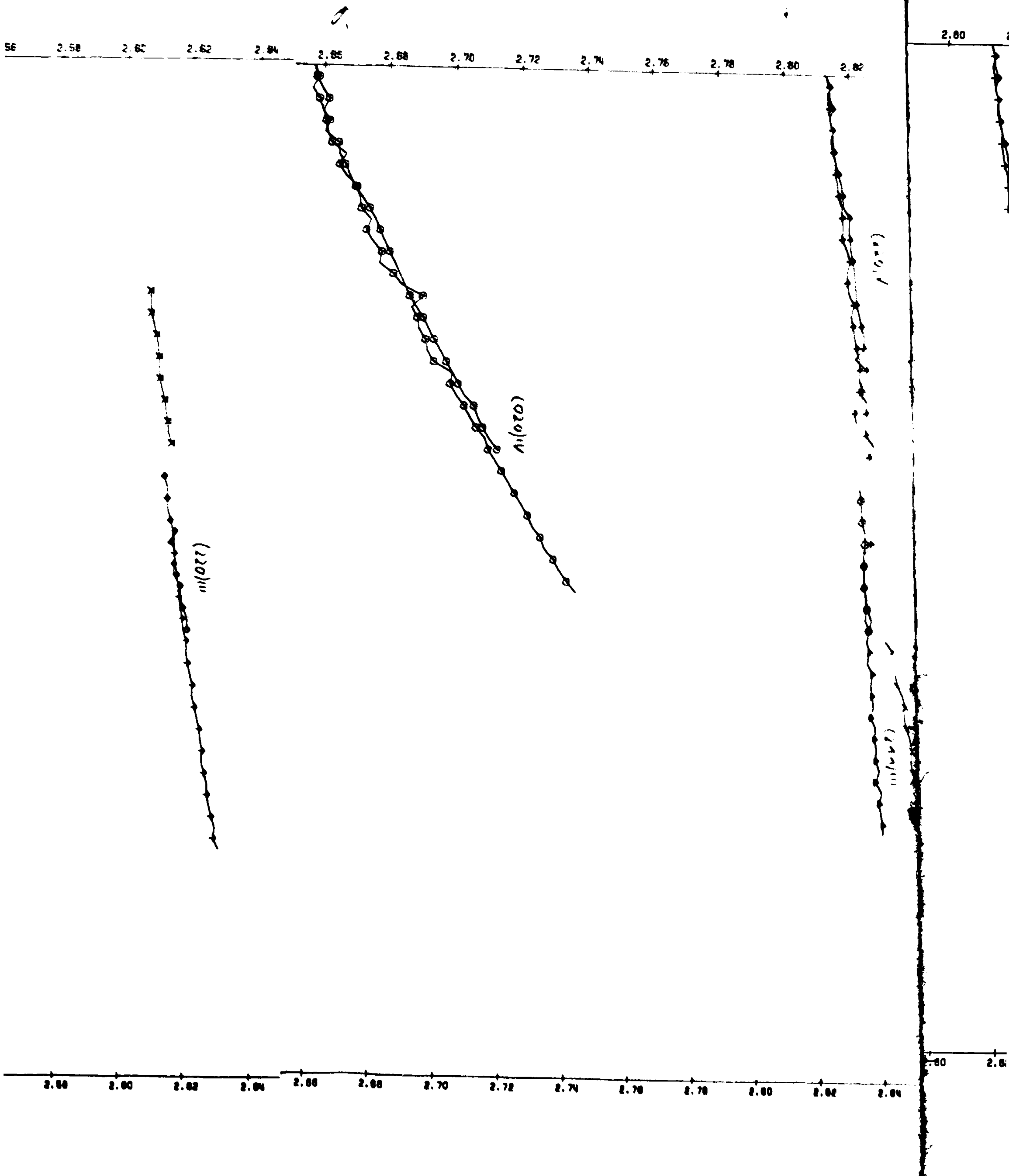

2
3.98 4.00 4.02 4.04 4.06 4.08 4.10 4.12 4.32 4.34 4.36 4.38 4.40 4.42
4.38 4.40
(100)V
(110)II
(100)I
3.98 4.00 4.02 4.04 4.06 4.08 4.10 4.12 4.32 4.34 4.36 4.38 4.40 4.42
38 4.40

3

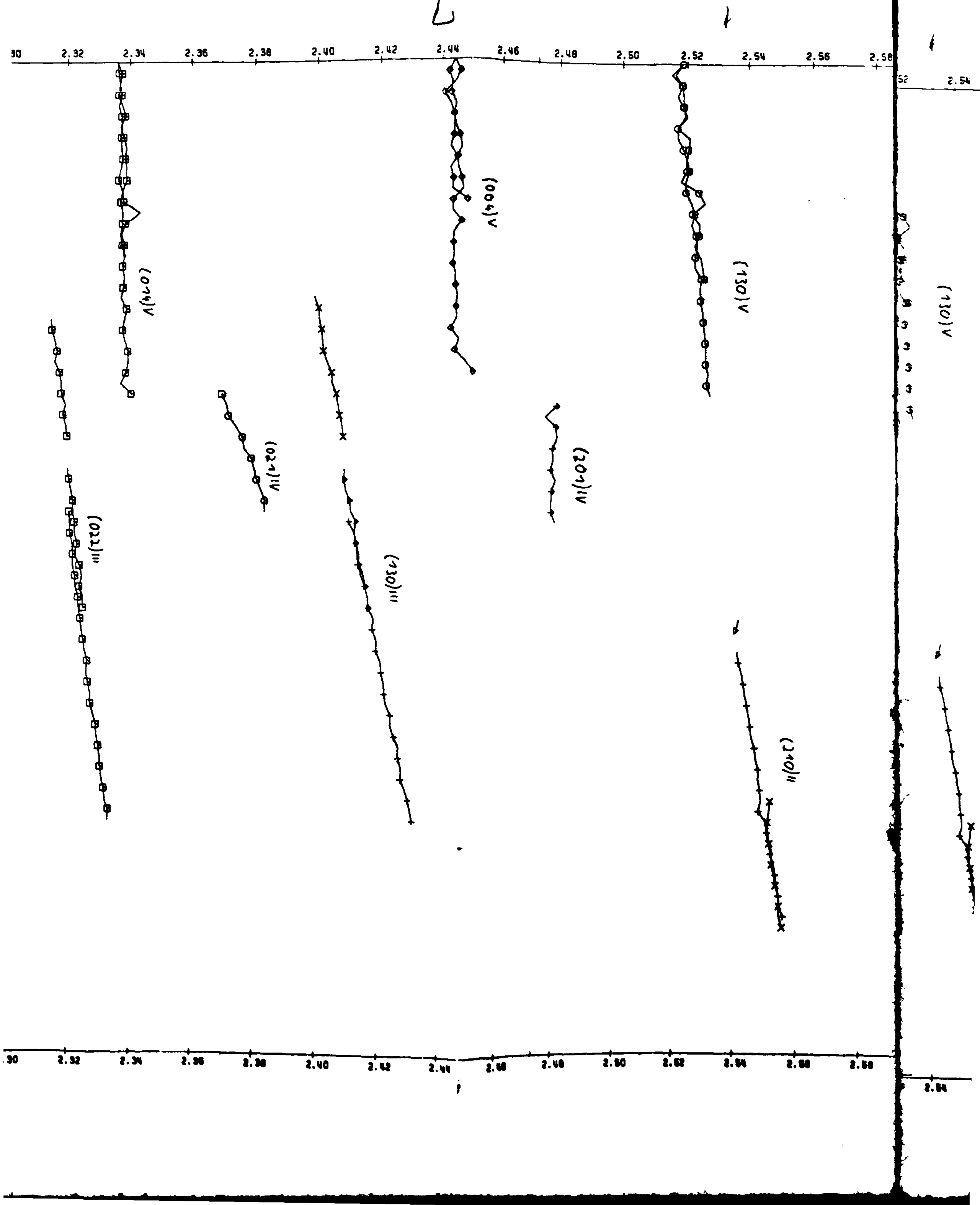

3.36
3.38
3.40
3.42
3.44
3.46
3.48
386
3.88
3.90
3.92
3.94
3.96
3.98
3.94
(110)IV
(110)IV
(111)III
(120)III

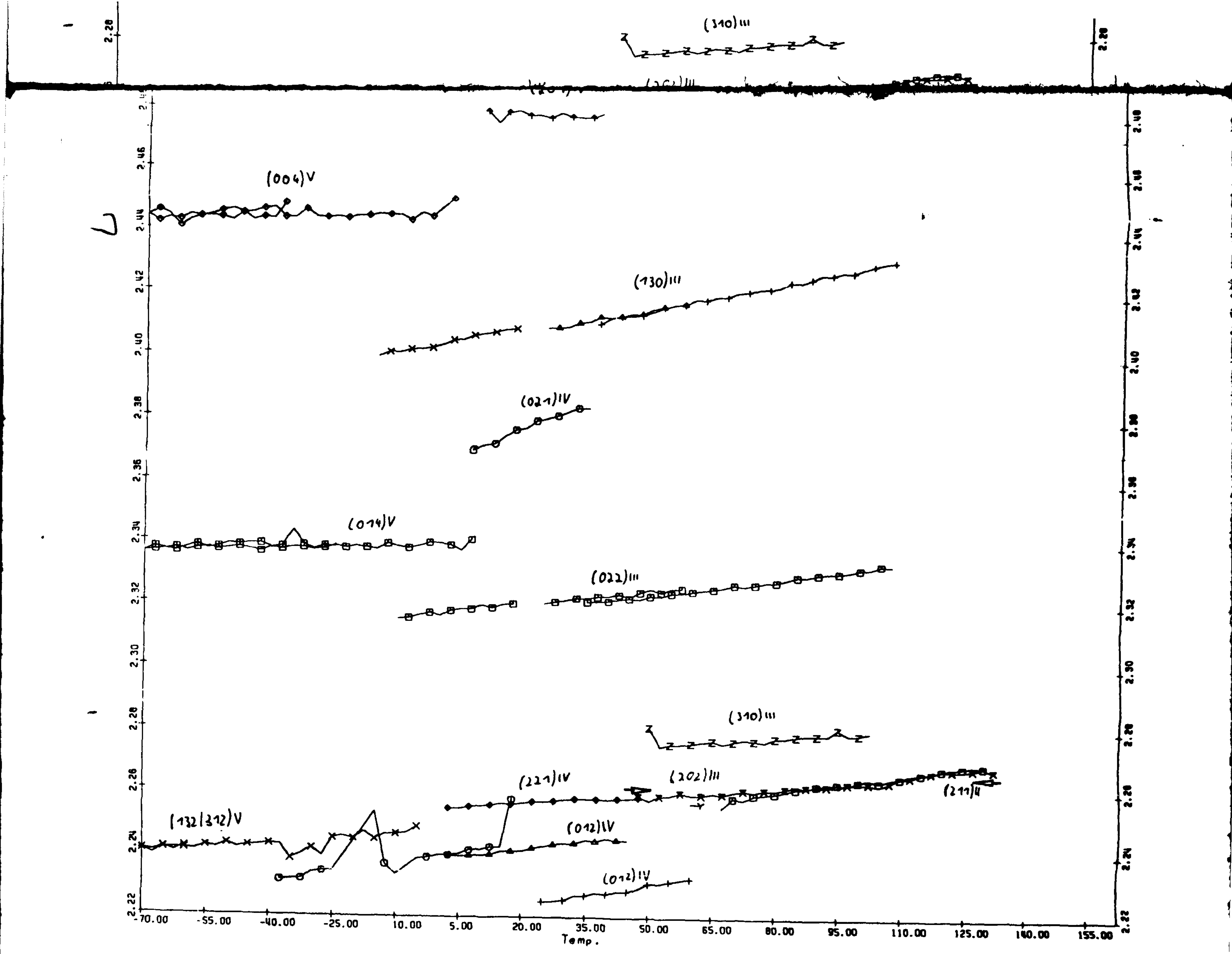

3.10
3.12
3.14
3.16
3.18
3.20
3.22
3.24
3.26
3.28
3.30
3.32
3.34
3.36
3.92
(023)V
III(120)
III(210)
N(111)





2.56
2.58
2.60
2.62
2.64
2.66
2.68
2.70
2.72
2.74
2.76
2.78
2.80
2.82
2.84


2.30
2.32
2.34
2.36
2.38
2.40
2.42
2.44
2.46
2.48
2.50
2.52
2.54
2.56
2.58

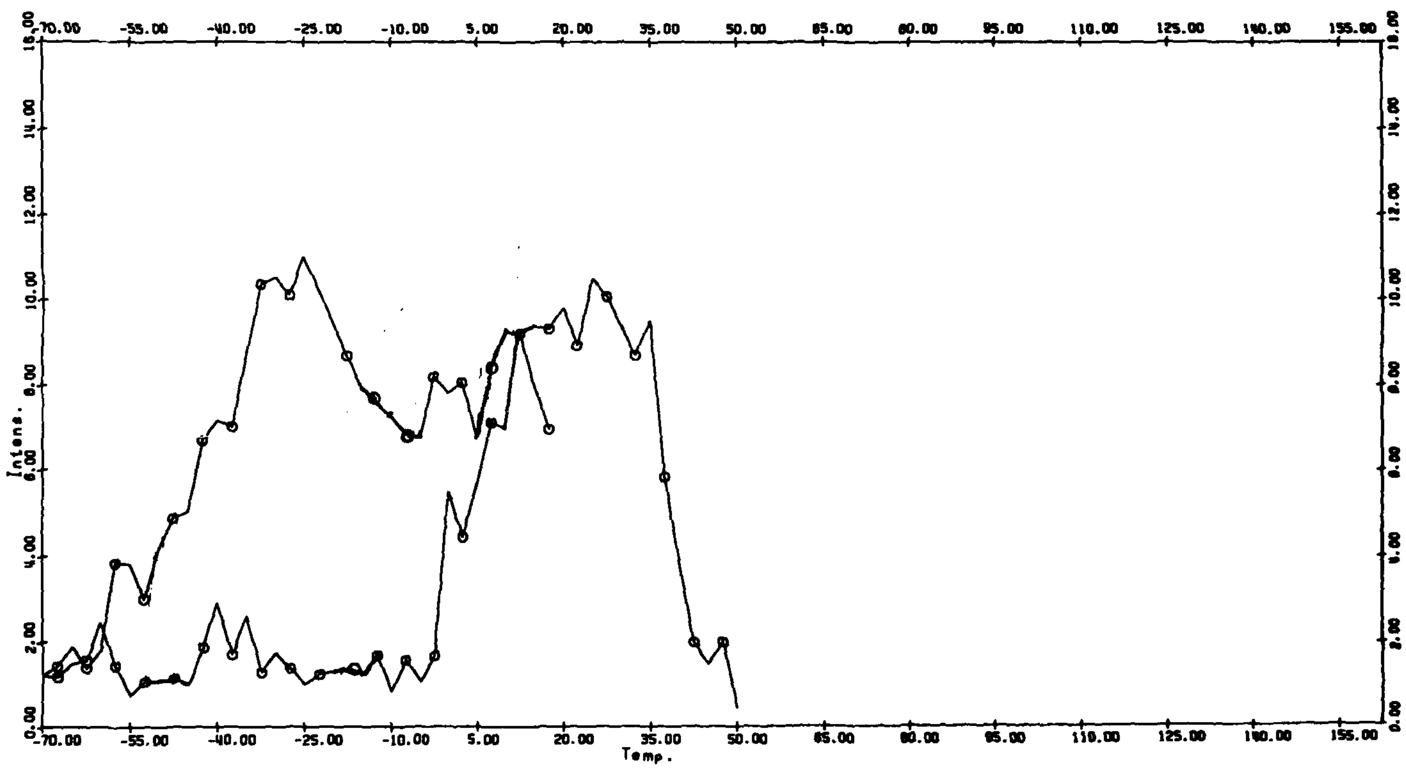


(004)V
(130)III
(021)IV
(014)V
(022)III
(310)III
(221)IV
(202)III
(211)II
(132|312)V
(012)IV
(012)IV
Temp.
-70.00
-55.00
-40.00
-25.00
10.00
5.00
20.00
35.00
50.00
65.00
80.00
95.00
110.00
125.00
140.00
155.00
2.22
2.24
2.26
2.28
2.30
2.32
2.34
2.36
2.38
2.40
2.42
2.44
2.46
2.48

KF 290388
201-70/150/20

(020)IV

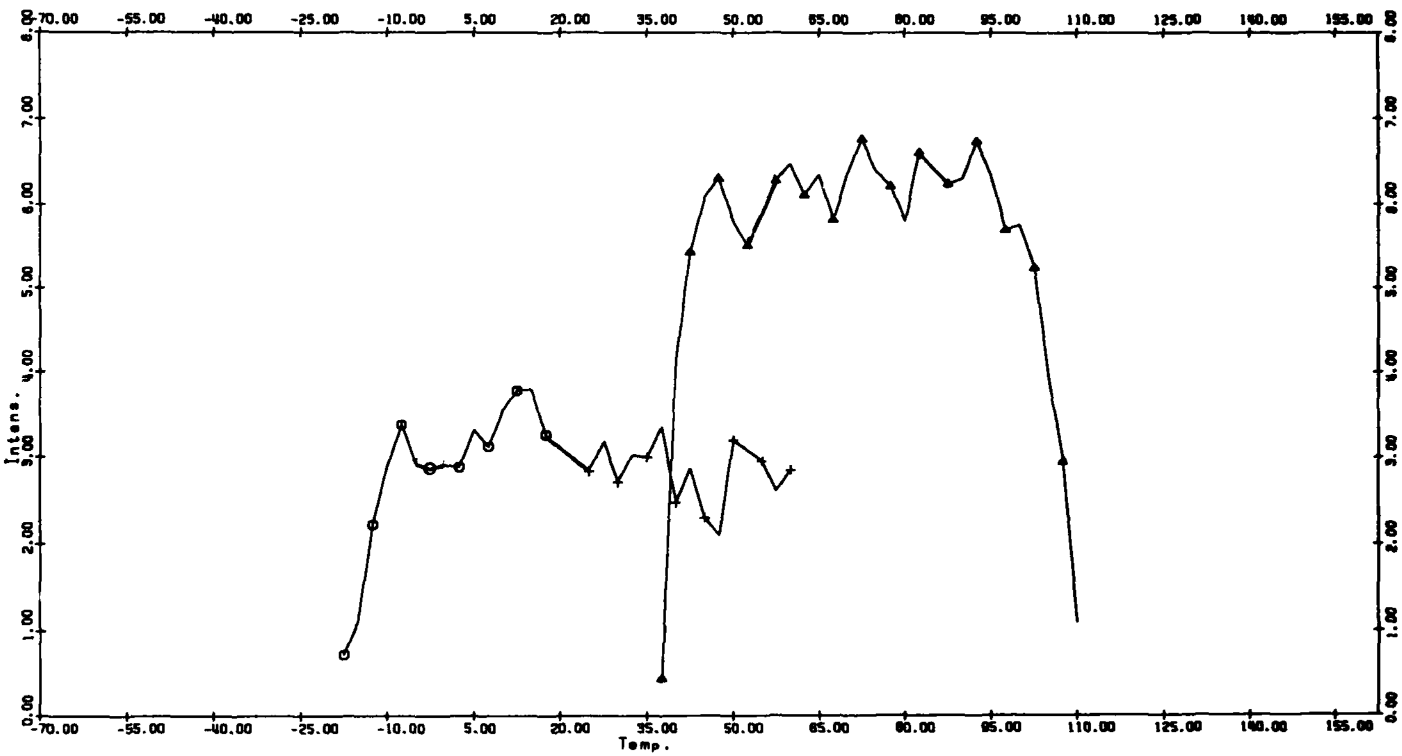
Intensities



K7 290388
20|-70/150|20

(220) III

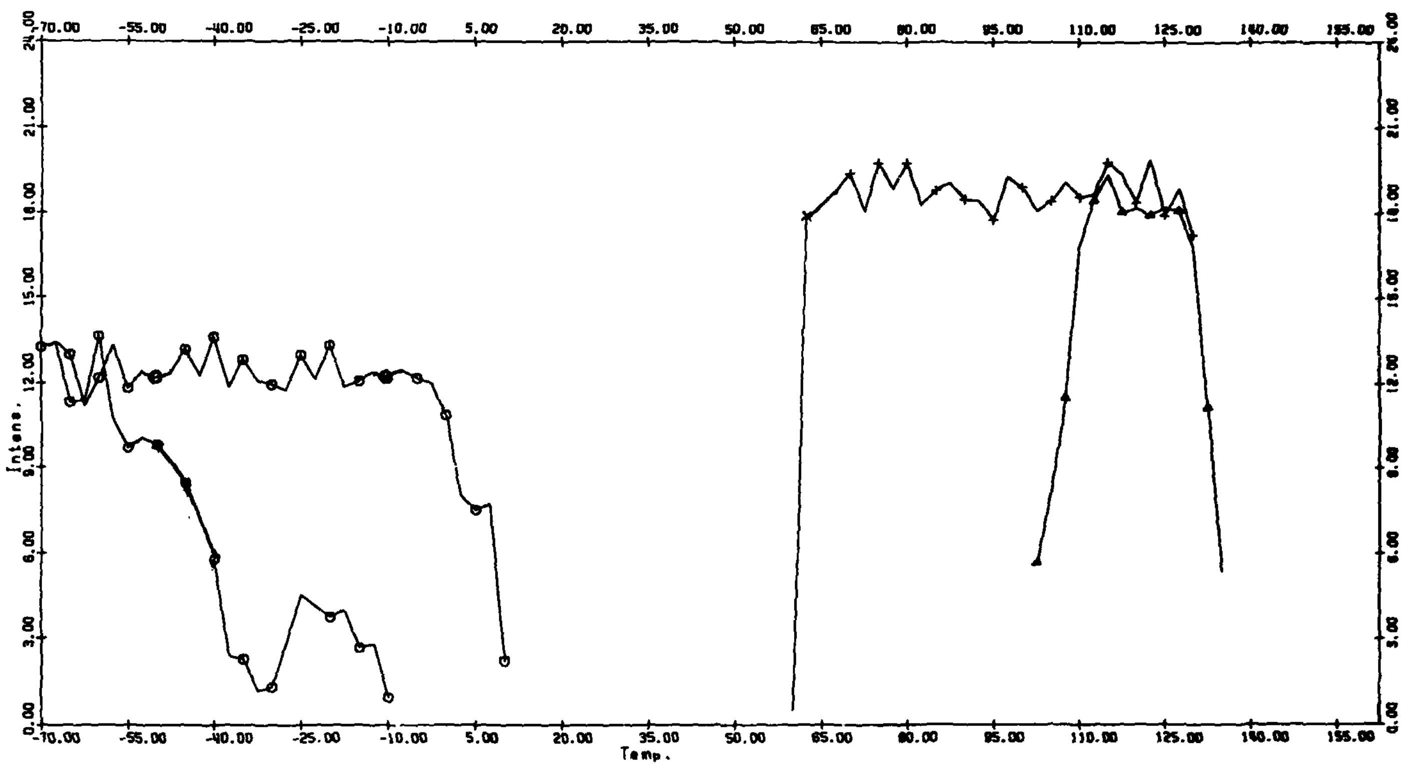
Intensities



KT 290388
20|-70|150|20

(022) V
(111) II

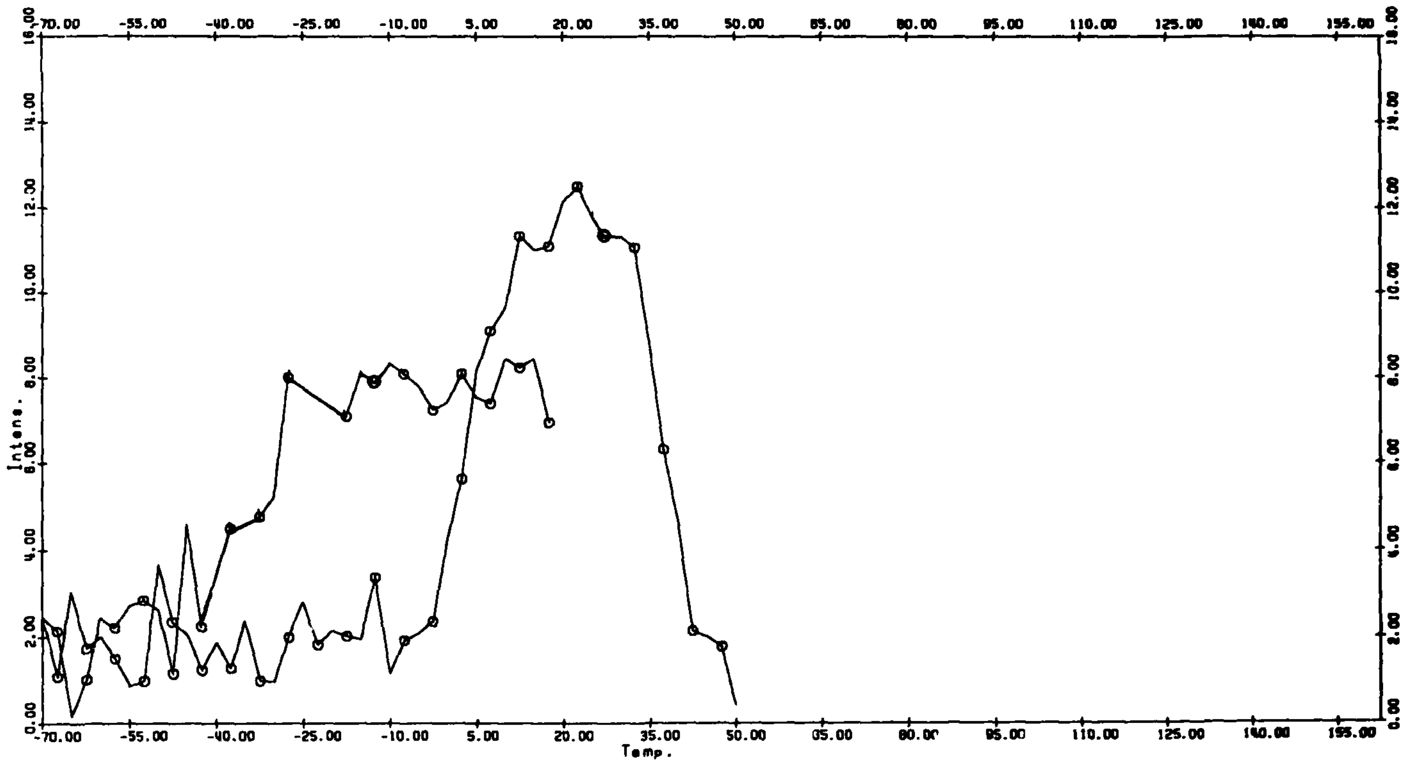
Intensities



KF290388
20|-70|150|20

(111)IV

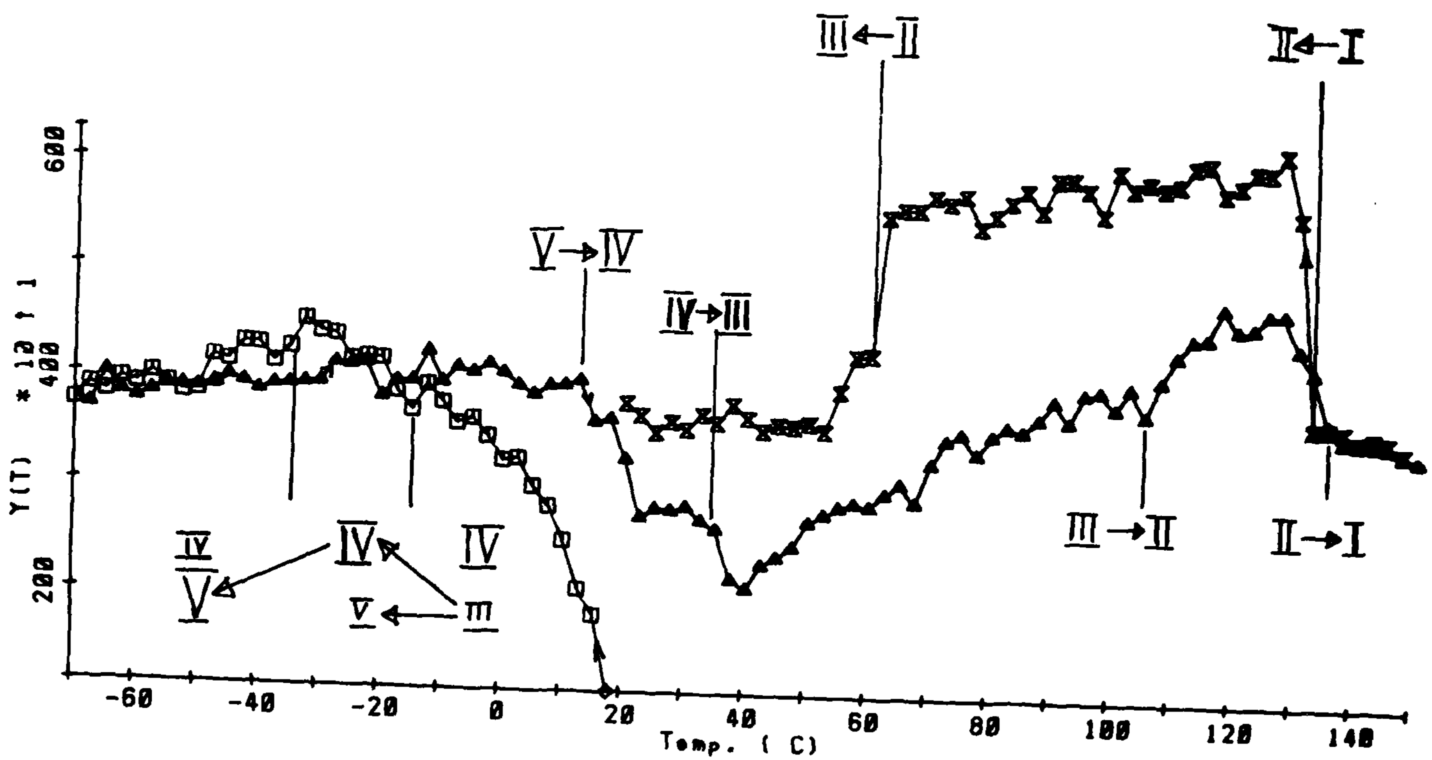
Intensities



Difference Curve

20|-70|150|20

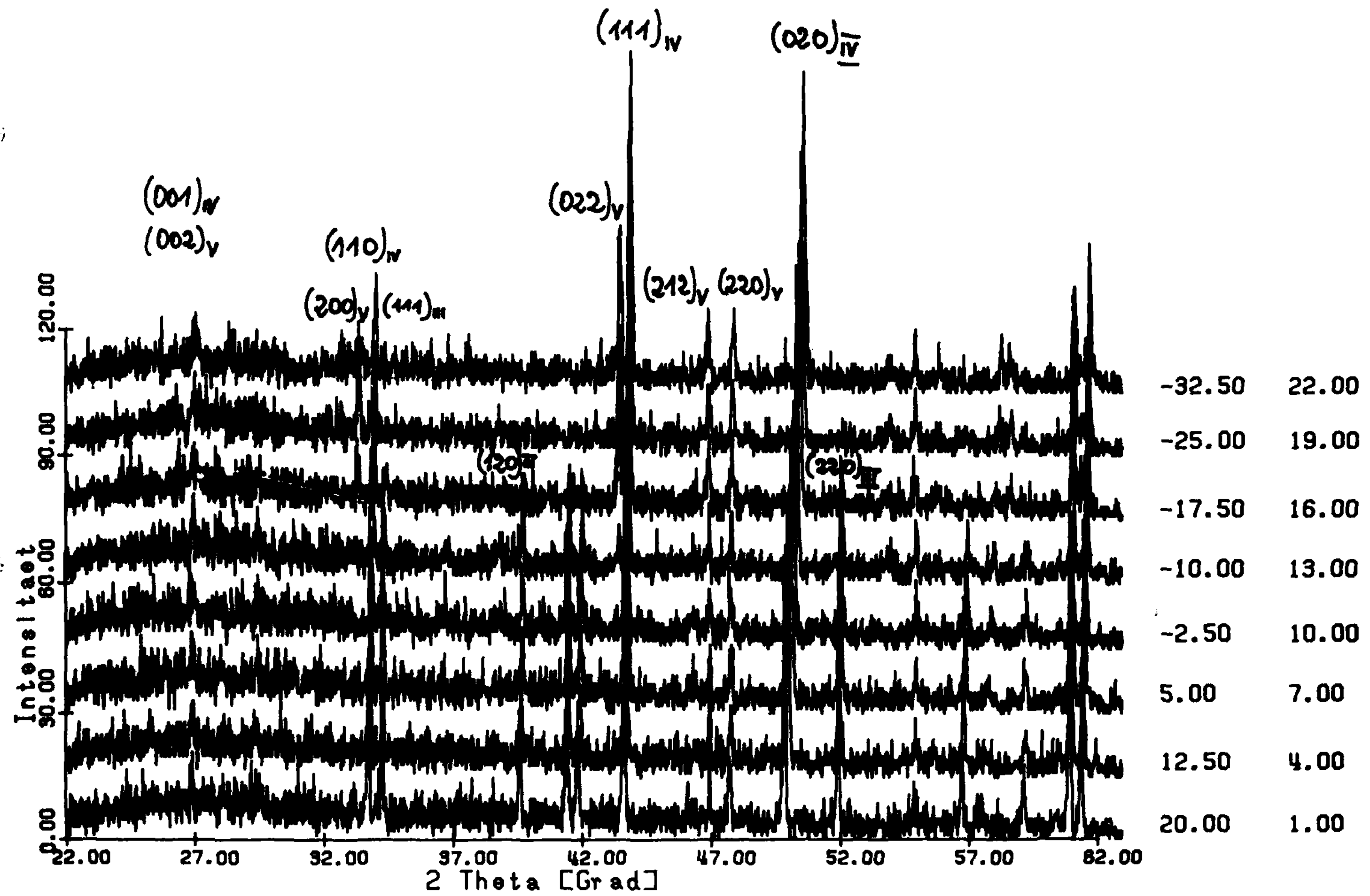
KF290388
Y(T)

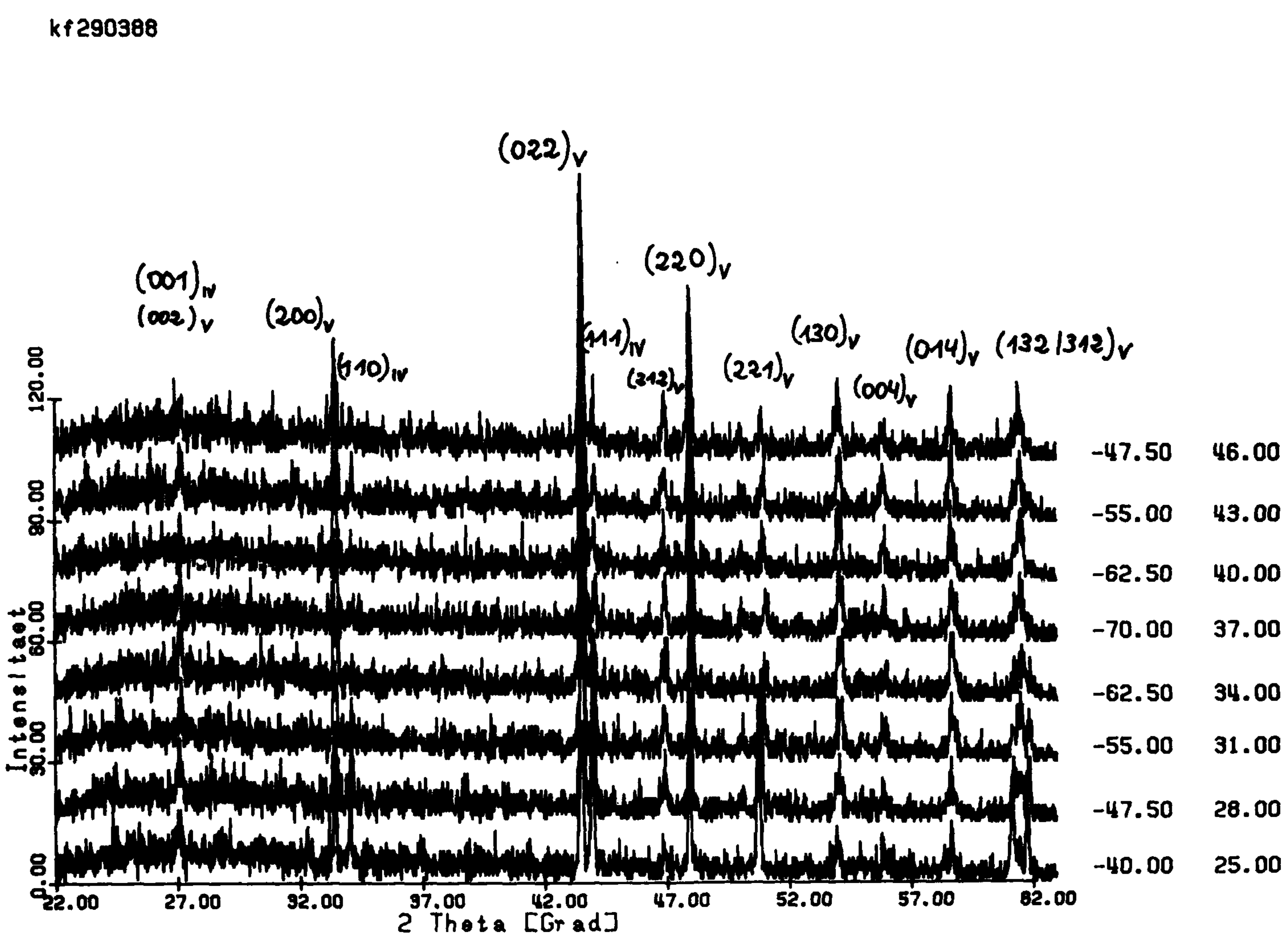


Diffraction Patterns

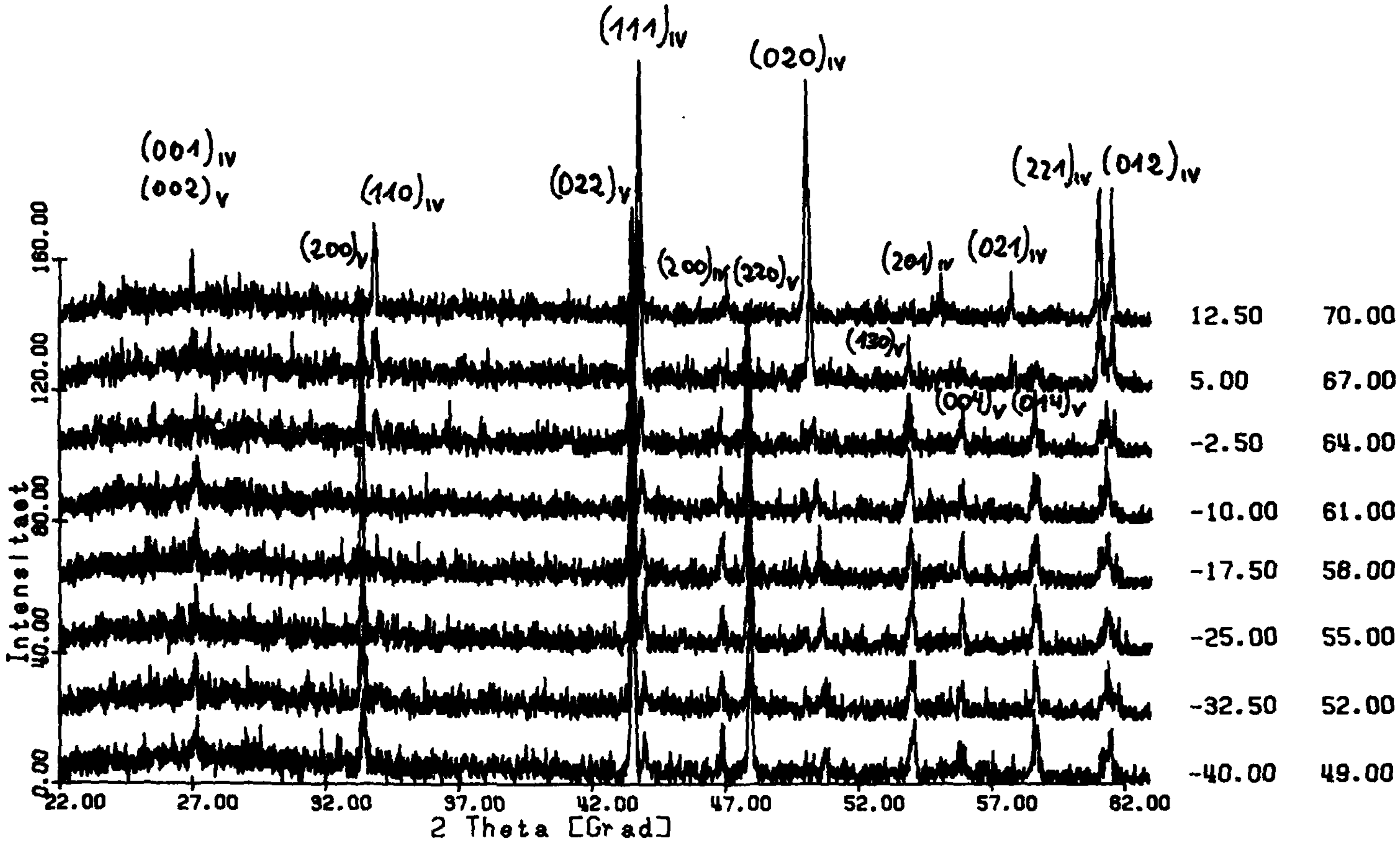
kf290388

20 / -70 / 150 / 20 °C



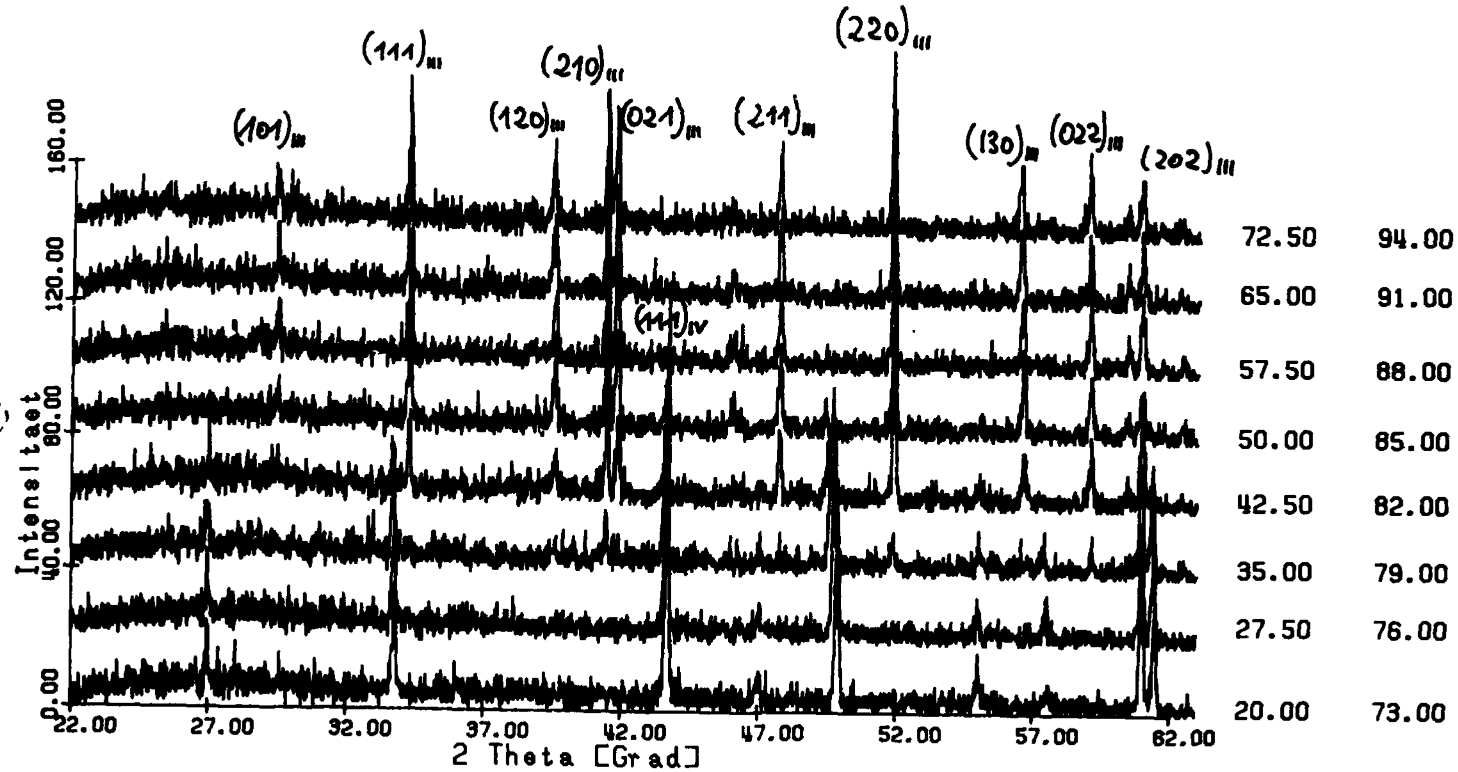

kf290388
(022)v
(220)v
(001)IV
(002)v
(200)v
(110)IV
(111)IV
(312)v
(221)v
(130)v
(004)v
(014)v
(132/312)v
120.00
90.00
60.00
30.00
0.00
Intensitaet
22.00
27.00
32.00
37.00
42.00
47.00
52.00
57.00
62.00
2 Theta [Grad]
-47.50 46.00
-55.00 43.00
-62.50 40.00
-70.00 37.00
-62.50 34.00
-55.00 31.00
-47.50 28.00
-40.00 25.00

kf290388



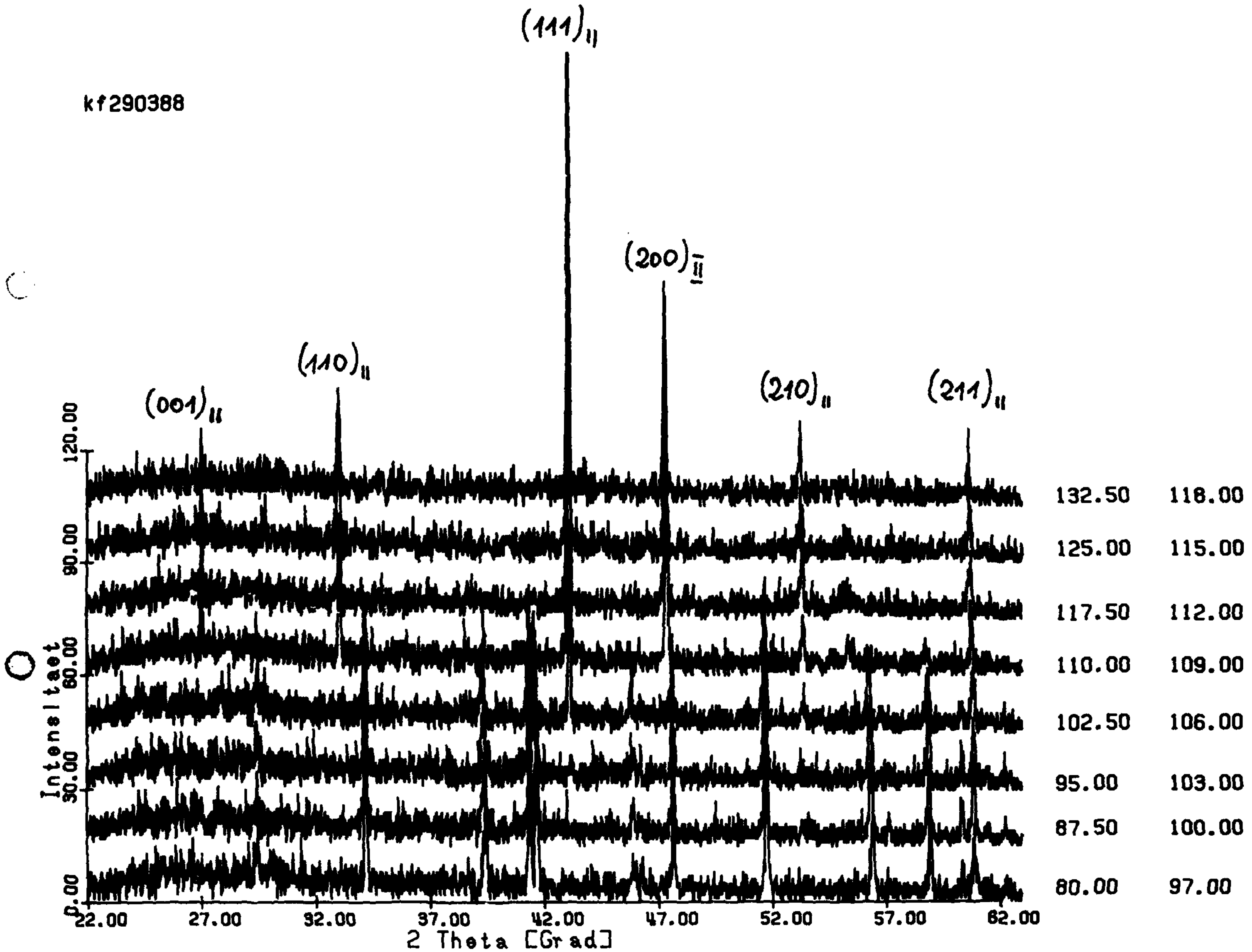
$(001)_{IV}$
$(002)_{V}$
$(200)_{V}$
$(110)_{IV}$
$(022)_{V}$
$(111)_{IV}$
$(200)_{IV}$
$(220)_{V}$
$(020)_{IV}$
$(130)_{V}$
$(201)_{IV}$
$(004)_{V}$
$(021)_{IV}$
$(014)_{V}$
$(221)_{IV}$
$(012)_{IV}$
Intensitaet
160.00
120.00
80.00
40.00
0.00
22.00
27.00
32.00
37.00
42.00
47.00
52.00
57.00
62.00
2 Theta [Grad]
12.50 70.00
5.00 67.00
-2.50 64.00
-10.00 61.00
-17.50 58.00
-25.00 55.00
-32.50 52.00
-40.00 49.00

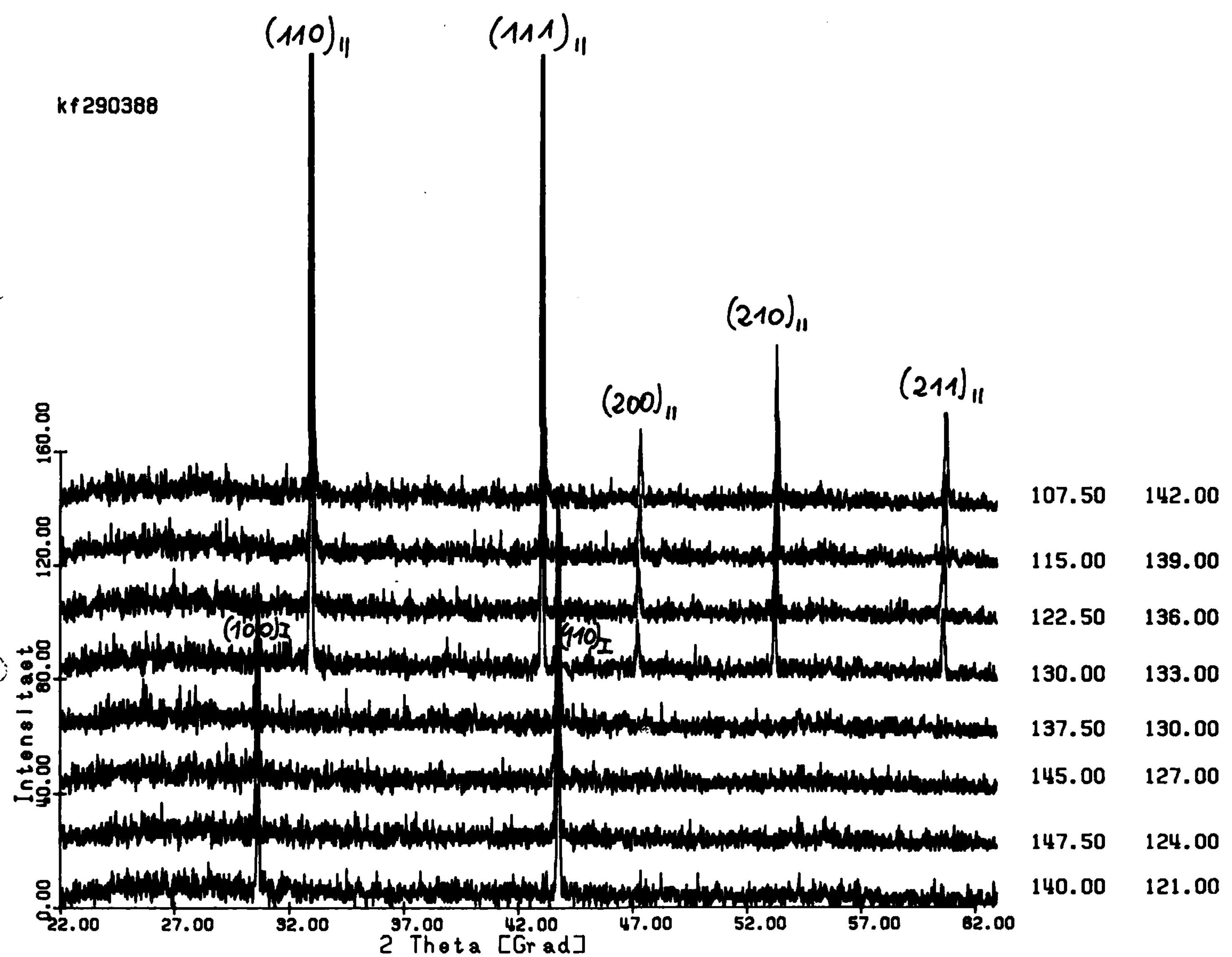
kf290388



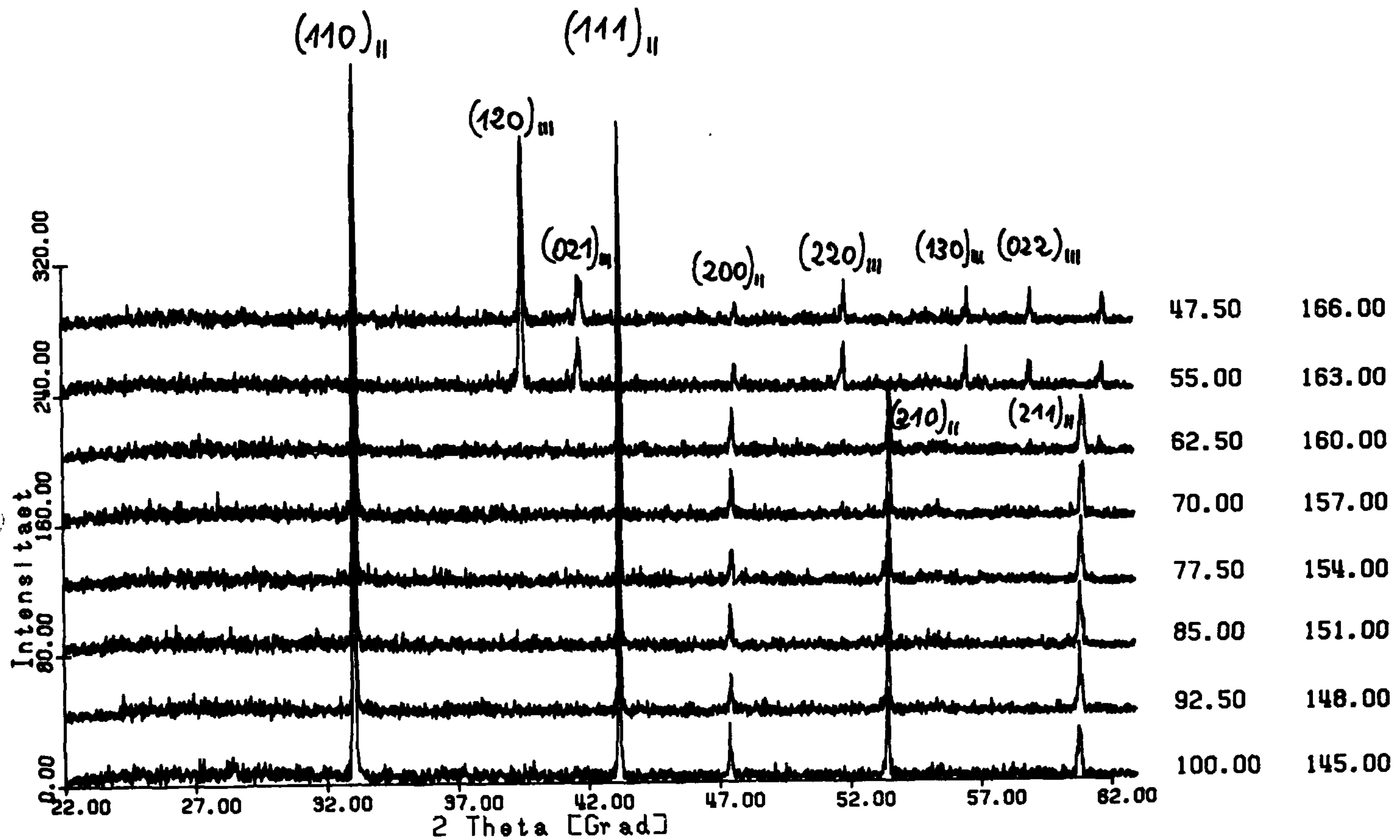
(101)III
(111)III
(120)III
(210)III
(021)III
(111)IV
(211)III
(220)III
(130)III
(022)III
(202)III
Intensitaet
0.00
40.00
80.00
120.00
160.00
22.00
27.00
32.00
37.00
42.00
47.00
52.00
57.00
62.00
2 Theta [Grad]
72.50 94.00
65.00 91.00
57.50 88.00
50.00 85.00
42.50 82.00
35.00 79.00
27.50 76.00
20.00 73.00

kf290388

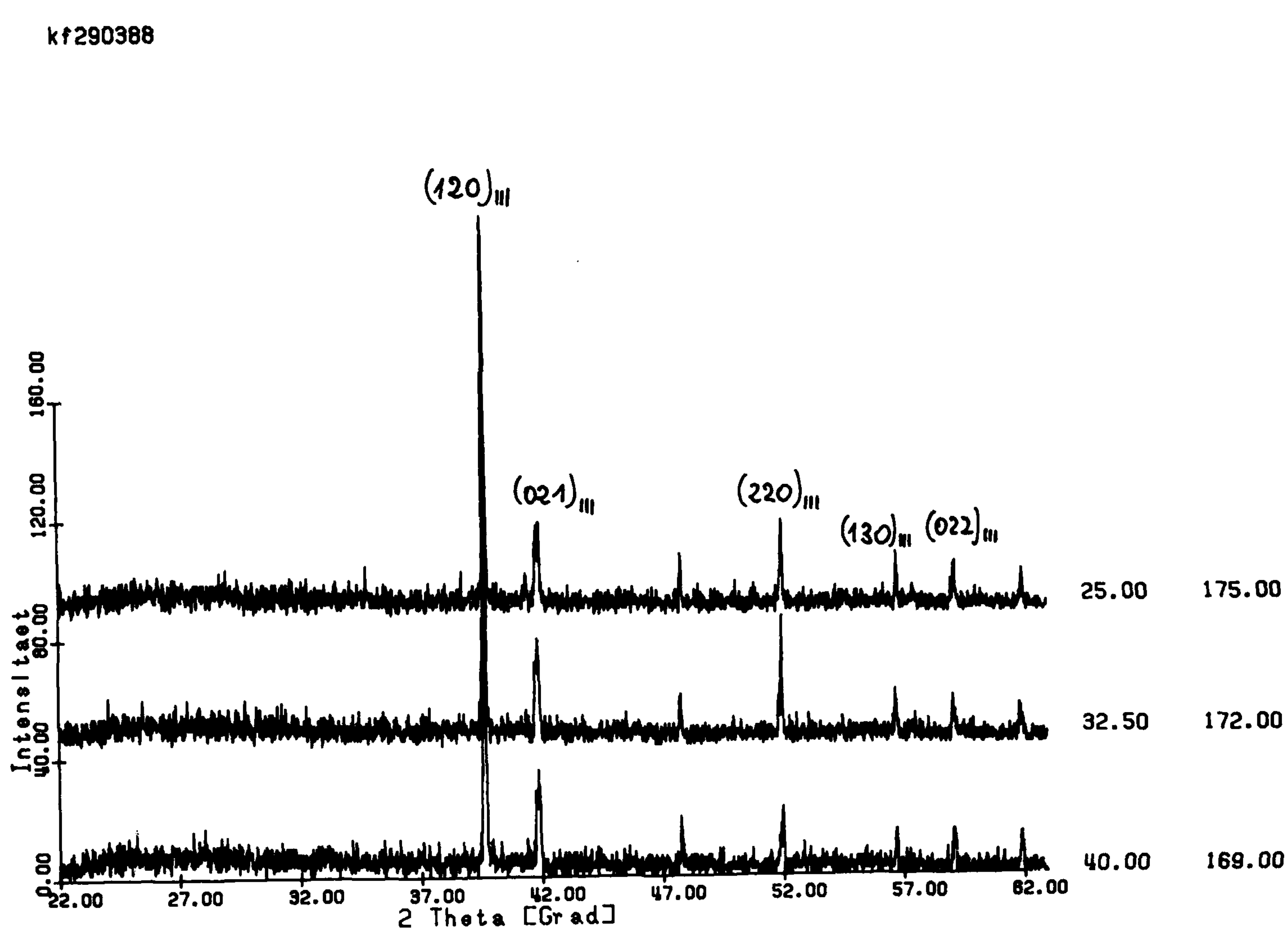

(111)II
(200)II
(110)II
(001)II
(210)II
(211)II
120.00
90.00
60.00
30.00
0.00
Intensitaet
22.00
27.00
32.00
37.00
42.00
47.00
52.00
57.00
62.00
2 Theta [Grad]
132.50 118.00
125.00 115.00
117.50 112.00
110.00 109.00
102.50 106.00
95.00 103.00
87.50 100.00
80.00 97.00


kf290388
(110)II
(111)II
(200)II
(210)II
(211)II
(100)I
(110)I
Intensitaet
0.00
40.00
80.00
120.00
160.00
22.00
27.00
32.00
37.00
42.00
47.00
52.00
57.00
62.00
2 Theta [Grad]
107.50 142.00
115.00 139.00
122.50 136.00
130.00 133.00
137.50 130.00
145.00 127.00
147.50 124.00
140.00 121.00

kf290388



(110)II
(111)II
(120)III
(021)III
(200)II
(220)III
(130)III
(022)III
(210)II
(211)II
47.50 166.00
55.00 163.00
62.50 160.00
70.00 157.00
77.50 154.00
85.00 151.00
92.50 148.00
100.00 145.00
Intensitaet
0.00
80.00
160.00
240.00
320.00
22.00
27.00
32.00
37.00
42.00
47.00
52.00
57.00
62.00
2 Theta [Grad]


kf290388
(120)III
(021)III
(220)III
(130)III
(022)III
25.00 175.00
32.50 172.00
40.00 169.00
160.00
120.00
80.00
40.00
0.00
Intensitaet
22.00
27.00
32.00
37.00
42.00
47.00
52.00
57.00
62.00
2 Theta [Grad]